

US EPA ARCHIVE DOCUMENT

**FINAL
TITANIUM DIOXIDE LISTING BACKGROUND
DOCUMENT
FOR THE INORGANIC CHEMICAL LISTING
DETERMINATION**

October 2001

U.S. ENVIRONMENTAL PROTECTION AGENCY
1200 PENNSYLVANIA AVENUE, NW
WASHINGTON, D.C. 20460

NOTE:

This document has been revised from the version provided in the docket for the proposed rule to reflect the Bevill exempt status of the vanadium recycle stream

TABLE OF CONTENTS

1. SECTOR OVERVIEW 1

 1.1 SECTOR DEFINITION, FACILITY NAMES AND LOCATION 1

 1.2 PRODUCTS, PRODUCT USAGE AND MARKETS 2

 1.3 PRODUCTION CAPACITY 4

 1.4 PRODUCTION, PRODUCT AND PROCESS TRENDS 4

2. DESCRIPTION OF MANUFACTURING PROCESSES 6

 2.1 PRODUCTION PROCESS DESCRIPTION 6

 2.2 PRODUCTION TRENDS, CHANGES AND IMPROVEMENTS 7

3. TITANIUM DIOXIDE WASTE CHARACTERIZATION, GENERATION, MANAGEMENT, SCREENING AND ASSESSMENT 9

 3.1 CHARACTERIZATION OF TITANIUM DIOXIDE WASTES 12

 3.2 EVALUATION OF TITANIUM DIOXIDE WASTE CATEGORIES 13

 3.2.1 Commingled Wastewaters from the Chloride Process, Including Wastewaters from Coke and Ore Recovery, Scrubber Water, Finishing Wastewaters and Sludge Supernatants 13

 3.2.2 Various Sands from Oxidation, Milling, and Scouring 20

 3.2.3 Gypsum from the Sulfate Process 23

 3.2.4 Digestion Scrubber Water from the Sulfate Process 29

 3.2.5 Sulfate Process Digestion Sludge 31

 3.2.6 Commingled Wastewaters from the Chloride and Sulfate Process 34

 3.2.7 Wastewater Treatment Sludges from Commingled Chloride and Sulfate Process Wastewaters 38

 3.2.8 Waste Acid (Ferric Chloride) from the Chloride-Ilmenite Process 44

 3.2.9 Non-Bevill-exempt Nonwastewaters from the Chloride-Ilmenite Process 53

 3.2.10 HCl from Reaction Scrubber, Chloride-Ilmenite Process 64

 3.2.11 Commingled Wastewaters from Chloride-Ilmenite Process 64

 3.2.12 Aluminum-containing Additive Vent Filters Solids from Chloride-Ilmenite Process 69

 3.2.13 Off-specification Titanium Dioxide Product 69

 3.2.14 Railcar/Trailer Product Washout 71

 3.3 OUT OF SCOPE WASTE 72

 3.3.1 Bevill-exempt Wastes 72

 3.3.2 Debris and Non-Process Wastes 77

 3.3.3 National Pollutant and Discharge System (NPDES) 77

 3.3 FORMATION OF DIOXINS/FURANS IN CHLORINATOR 78

Appendix A: Summary of Analytical Data Results

Appendix B: Split Sample Results

LIST OF TABLES

Table 1.1 - Titanium Dioxide Producers 1

Table 1.2 - Titanium Dioxide Production Capacity 4

Table 3.1 - Waste Reported by Titanium Dioxide Facilities Using the Chloride Process 10

Table 3.2 - Waste Reported by Titanium Dioxide Facilities Using the Sulfate Process 10

Table 3.3 - Waste Reported by Titanium Dioxide Facilities Using the Chloride-Ilmenite Process
..... 11

Table 3.4 - Commingled Wastewaters from the Chloride Process 15

Table 3.5 - Initial Screening Analysis for Commingled Wastewaters from Chloride Process .. 18

Table 3.6 - Waste Management Practices and Volumes for Various Sands from Oxidation, Milling,
and Scouring 21

Table 3.7 - Initial Screening Analysis for Milling Sand 21

Table 3.8 - Initial Screening Analysis for Scouring Sand 23

Table 3.9 - Waste Management Practices and Volumes for Gypsum from Sulfate Process 24

Table 3.10 - Initial Screening Analysis for Primary and Secondary Gypsum 26

Table 3.11 - Waste Management Practices and Volumes for Digestion Scrubber Water from the
Sulfate Process 30

Table 3.12 - Initial Screening Analysis for Digestion Scrubber Water from the Sulfate Process
..... 31

Table 3.13 - Waste Management Practices and Volumes for Sulfate Process Digestion Sludge
..... 32

Table 3.14 - Initial Screening Analysis for Sulfate Process Digestion Sludge 34

Table 3.15 - Commingled Wastewaters from the Chloride and Sulfate Process 36

Table 3.16 - Initial Screening Analysis for Commingled Wastewaters from the Chloride and
Sulfate Process 37

Table 3.17 - Estimation of Non-Exempt Solids Contribution to Wastewater Treatment Sludges
from Commingled Chloride and Sulfate Process Wastewaters at Millennium Baltimore
..... 39

Table 3.18 - Waste Management Practices and Volumes for Wastewater Treatment Sludges from
Commingled Chloride and Sulfate Process Wastewaters 40

Table 3.19 - Initial Screening Analysis for Wastewater Treatment Sludges from Commingled
Chloride and Sulfate Process Wastewaters 42

Table 3.20 - Waste Management Practices and Volumes for Waste Acid (ferric chloride) from the
Chloride-Ilmenite Process 45

Table 3.21 - Summary of Analytical Results for Waste Acid (ferric chloride) from the Chloride-
Ilmenite Process 48

Table 3.22 - Summary of Analytical Results for Ferric Carbonate 52

Table 3.23 - Estimation of Non-Bevill Exempt Solids Contribution to DuPont Edge Moor's
Wastewater Treatment Solids 55

Table 3.24 - Estimate of Non-Bevill Exempt Solids Contribution to DuPont New Johnsonville's
Wastewater Treatment Solids 56

Table 3.25 - Estimation of Non-Bevill Exempt Solids Contribution to DuPont DeLisle's
Wastewater Treatment Solids 58

Table 3.26 - Waste Management Practices and Volumes for Non-Bevill-exempt Nonwastewaters

from the Chloride-Ilmenite Process 59

Table 3.27 - Initial Screening Analysis for Non-Bevill-exempt Nonwastewaters from the Chloride-Ilmenite Process 60

Table 3.28 - Comparison of Iron Rich® Total Analyses to Soil Screening Levels (SSL) 63

Table 3.29 - Volumes for Commingled Wastewaters from Chloride-Ilmenite Process 66

Table 3.30 - Initial Screening Analysis for Commingled Wastewaters from Chloride-Ilmenite Process 68

Table 3.31 - Volumes of Off-specification Titanium Dioxide Product 71

Table 3.32 - Initial Screening Analysis for Off-specification Titanium Dioxide Product 72

Table 3.33 - Initial Screening Analysis for Railcar/Trailer Product Washout 73

Table 3.34 - Bevill-exempt-Waste Solids from Titanium Tetrachloride Production Via the Chloride Process 75

Table 3.35 - Bevill-exempt Waste Solids from Titanium Tetrachloride Production via the Chloride-Ilmenite Process 77

Table 3.36 - Bevill-exempt Storage and Handling Wastes 77

Table 3.37 - Debris and Non-Process Waste 78

Table 3.38 - Permitted NPDES Waste 79

LIST OF FIGURES

Figure 1.1 - Geographical Distribution of Titanium Dioxide Producers 3

Figure 2.1 - Process Flow Diagram for the Production of Titanium Dioxide 8

1. SECTOR OVERVIEW

1.1 SECTOR DEFINITION, FACILITY NAMES AND LOCATION

Titanium dioxide is produced in the United States by 9 manufacturers through the chloride, sulfate, or the chloride-ilmenite processes (See Section 2). Cerac, Inc. located in Milwaukee, WI reported producing titanium dioxide but was not considered as part of this listing determination because the facility is a specialty products manufacturer of many chemicals, including high purity titanium dioxide in very small amounts (16.2 kg./yr) for laboratories and the research community.

Table 1.1 lists the names and locations of the nine manufacturers and their respective type. **Figure 1.1** shows the geographical location of the facilities listed in Table 1.1.

Table 1.1 - Titanium Dioxide Producers		
Facility Name	Facility Location	Production Process
1. Kemira Pigments, Inc. ¹	One Kemira Road PO Box 368 Savannah, GA 31402	Sulfate
		Chloride
2. Millennium Inorganic Chemicals Inc. (formerly SCM)	3901 Fort Armistead Road Baltimore, MD 21226	Sulfate
		Chloride
3. Kerr-McGee Chemical Corp.	40034 Kerr-McGee Road Hamilton, MS 39746	Chloride
4. Kronos/Louisiana Pigment Co.	3300 Bayou Dinde Road West Lake, LA 70669	Chloride
5. Millennium Inorganic Chemicals, Inc. Plant I	2900 Middle Road PO Box 310 Ashtabula, OH 44004	Chloride
6. Millennium Inorganic Chemicals, Inc. Plant II	2426 Middle Road PO Box 160 Ashtabula, OH 44004	Chloride
7. E.I. DuPont de Nemours & Co., DeLisle Plant	7685 Kiln-DeLisle Road PO Box 430 Pass Christian, MS 39571	Chloride-Ilmenite
8. E.I. DuPont de Nemours & Co., Edge Moor Plant	4600 Hay Road (Shipping) 104 Hay Rd. (Mailing) Edge Moor, DE 19809	Chloride-Ilmenite

¹Kerr-McGee acquired Kemira's TiO₂ facilities in Savannah, GA; ChemExpo; May 22, 2000.

Table 1.1 - Titanium Dioxide Producers

Facility Name	Facility Location	Production Process
9. E.I. DuPont de Nemours & Co., New Johnsonville Plant	One DuPont Rd. PO Box 2194 New Johnsonville, TN	Chloride-Ilmenite

1.2 PRODUCTS, PRODUCT USAGE AND MARKETS

Titanium dioxide has the molecular formula TiO_2 , a molecular weight of 79.90, and, when used as a pigment commonly is referred to as Pigment White 6 (Colour Index Number 77891). Titanium dioxide is a colorless solid at room temperature, melts at 1830 °C, and boils between 2500 and 3000 °C.²

More than 50 percent of the titanium dioxide produced is used in paints, varnishes and lacquer. In paints, titanium dioxide is used primarily to whiten and opacify polymeric binder systems. Even medium to deep shades usually contain some titanium dioxide. It also is used in coatings where exterior durability is needed.³

More than 25 percent of the titanium dioxide produced is used in paper and paperboard. The paper industry uses titanium dioxide in two different applications: as a direct addition to whiten and opacify the paper stock, and in the manufacture of coatings that are applied to the paper product.

Titanium dioxide is used in plastics to impart whiteness and opacity.⁴ Approximately 20 percent of the titanium dioxide produced is used in plastics to impart whiteness and opacity.⁵

Titanium dioxide also is used in the manufacture of many other products including printing inks, rubber, floor coverings, ceramics, food and pharmaceuticals.⁶

²ECDIN Home Page, http://ecdin.etomep.net/cgi-bin_ecd

³ChemExpo Home Page, <http://www.chemexpo.com/news/profile970912.cfm>

⁴Ibid

⁵Ibid

⁶ChemExpo Home Page, <http://www.chemexpo.com/news/profile970912.cfm>

Figure 1.1 - Geographical Distribution of Titanium Dioxide Producers¹



¹ See **Table 1.1** for facility name and location.

1.3 PRODUCTION CAPACITY

In 1997 the maximum production capacity in the United States was approximately 1,405,000 metric tons per year (MT/yr).⁷ **Table 1.2** provides the list of titanium dioxide production facilities and their reported capacities.

Production Facility	Production Process	Capacity (10³ MT/yr)
1. Kemira Pigments, Inc.	Sulfate	60
	Chloride	100
2. Millennium Inorganic Chemicals (formerly SCM), Baltimore Plant	Sulfate	44
	Chloride	51
3. Kerr-McGee Chemical Corp.	Chloride	160
4. Kronos/Louisiana Pigment Co.	Chloride	110
5. Millennium Inorganic Chemicals, Inc., Ashtabula Plant I	Chloride	104
6. Millennium Inorganic Chemicals, Inc., Ashtabula Plant II	Chloride	86
7. E.I. DuPont de Nemours & Co., DeLisle Plant	Chloride-Ilmenite	250
8. E.I. DuPont de Nemours & Co., Edge Moor Plant	Chloride-Ilmenite	145
9. E.I. DuPont de Nemours & Co., New Johnsonville Plant	Chloride-Ilmenite	295
		1,405

1.4 PRODUCTION, PRODUCT AND PROCESS TRENDS

The 1997 data shows the demand for titanium dioxide as 1.175 million tons. The demand is projected to be 1.362 million tons in the year 2001.⁸

For the period between 1987 and 1996, titanium dioxide sales have grown 2 to 2.5 percent per year. A 2 to 4 percent annual growth is projected through the year 2001. The sale price for

⁷Ibid.

⁸ChemExpo Home Page, <http://www.chemexpo.com/news/profile970912.cfm>.

titanium dioxide was highest between the 1981 and 1996 at \$ 1.04 per pound. The 1997 reported data shows the price of titanium dioxide between \$0.92 to \$0.94 per pound.⁹

With the U.S. being a principal world producer, and limited foreign capacity, there has been leeway to raise world prices in the past years as demand increased. There is a limit to price elasticity, however, particularly in the paper industry, where competitive materials replace (or limit) the use of titanium dioxide in some applications. The paper industry is striving to reduce consumption of titanium dioxide because of the high price levels. This has been done, particularly in plants using alkaline paper making, by increasing calcium carbonate use as a titanium dioxide extender. Although more difficult to replace in paint applications, a reduction and rationalization is a possibility if prices continue to rise.¹⁰

The oldest production process for titanium dioxide is the sulfate process. The major difference between the chloride and the chloride-ilmenite process is the process feed stock. The two main titanium bearing minerals sources that are used as feedstock in the production of titanium dioxide are ilmenite and rutile. The most abundant titanium bearing mineral is ilmenite and is comprised of approximately 43 to 65 percent titanium dioxide. Synthetic rutile, from the acid leach of ilmenite, is the second major feedstock for titanium dioxide production and contains approximately 95 percent titanium dioxide. Titaniferrous slag, which is 70 to 80 percent titanium dioxide, is a co-product of smelting.¹¹ The chloride process produces a smaller quantity of waste materials than the sulfate process, but the chloride process is difficult to operate. The extreme corrosiveness of the high temperature chlorine employed in the process contributes to the difficulty. The oxidation step in the process is also extremely difficult to control due to burner configuration and product recovery. DuPont holds significant patent protection in a technology that addresses this fundamental problem with the oxidation step.¹²

⁹Ibid.

¹⁰54 FR 36592 (Sept. 1, 1989), 55 FR 2322 (Jan. 23, 1990), the July 31, 1990 Report to Congress on Wastes from Mineral Processing, and 56 FR 27300 (June 13, 1991).

¹¹Titanium Tetrachloride Production by the Chloride Ilmenite Process, Technical Background Document, Office of Solid Waste, U.S. EPA, April 1998.

¹²Ibid.

2. DESCRIPTION OF MANUFACTURING PROCESSES

2.1 PRODUCTION PROCESS DESCRIPTION

As noted above in Section 1, titanium dioxide is manufactured using three processes: the chloride, sulfate, and chloride-ilmenite processes. The following are general descriptions of these three production processes. **Figure 2.1** contains general process flow diagrams for the production of titanium dioxide via the chloride, sulfate and chloride-ilmenite processes. These descriptions and flow diagrams do not account for specific process variations reported by the titanium dioxide manufacturers.

Chloride process

The chloride process begins with the conversion of rutile or high-grade ilmenite into titanium tetrachloride (TiCl_4). This step occurs in a fluidized bed chlorinator in the presence of chlorine gas at a temperature of approximately 900°C . Petroleum coke also is added as a reductant. The volatile TiCl_4 , including other metal chlorides such as vanadium oxychloride, exit the chlorinator as overhead vapor. The non-volatile chlorides and the unreacted coke and ore solids are removed from the gas stream and from the bottom of the chlorinator. The gaseous product stream is purified to separate the titanium tetrachloride from other metal chloride impurities using condensation and chemical treatment. Vent gases from the chlorinator are scrubbed using water and caustic solutions prior to venting to the atmosphere. The purified TiCl_4 is then oxidized to TiO_2 , driving off chlorine gas, which is recycled to the chlorinator. The pure TiO_2 is slurried and sent to the finishing process which includes milling, addition of inorganic and organic surface treatments, and/or spray drying of the product TiO_2 . The product can be sold as a packaged dry solid or a water-based slurry.

Typical wastes generated by the chloride process includes wastewaters from chlorinator coke and ore solids recovery, reaction scrubbers, chemical tank storage scrubbers, product finishing operations and wastewater treatment solids decantation. Bevill-exempt waste solids are also generated during the production of titanium tetrachloride. Waste sands from finishing (milling) of the titanium dioxide product, scouring of oxidation process units, and blasting of reactor internal surfaces prior to replacement of refractory are also generated.

Sulfate process

The sulfate process starts with dried and milled slag being dissolved in sulfuric acid and water in a digester. This produces a titanyl sulfate liquor. From the digester the titanyl sulfate liquor goes to a clarification tank where the undissolved ore and solids are allowed to settle. The titanium liquor then is concentrated and hydrolyzed to titanium dioxide hydrate. The titanium dioxide hydrate precipitates from the ferrous sulfate and sulfuric acid and is separated through filtration. After filtration the hydrated titanium dioxide slurry is sent to a calciner, where the titanium dioxide crystals grow to their final crystalline size and residual water and H_2SO_4 are removed. The dried titanium dioxide is sent to pigments finishing. This finishing phase involves any required milling and or chemical treatment, such as surface coating with silica or alumina.

Typical wastes generated by the sulfate process includes digestion scrubber water from the scrubbing of gasses generated during the digestion step. Digestion sludge is generated after the filtering of the bottom solids from the settled titanyl sulfate liquor generated during digestion. A waste acid is generated as a result of the filtering of the titanium dioxide hydrate. This waste acid is used in the production of commercial gypsum. Other wastewaters are generated during the calcination and finishing steps of the process. Product milling sands is also generated during the finishing process.

Chloride-ilmenite process

In the chloride-ilmenite process, titanium-bearing ore is converted to titanium tetrachloride. As in the chloride process, the chloride-ilmenite process takes place in a chlorinator where the ore is chlorinated in the presence of coke as a reducing agent. The gaseous product stream is purified to separate the titanium tetrachloride from other metal chloride impurities, including ferric chloride (FeCl_3) which is present in higher concentrations than the chloride process because of the high iron content in the ore. The separation is done via condensation and chemical treatment. The process for converting TiCl_4 to TiO_2 is similar to that used in the chloride process, described above.

Typical wastes generated by the chloride-ilmenite process includes coke and ore solids (Bevill exempt) that remain unreacted during the chlorination process. A waste acid solution, usually called ferric or iron chloride waste acid, is also generated when the combined stream of unreacted coke and ore solids, metal chloride solids, and vanadium compounds is acidified using water or waste HCl from the reaction scrubber. Process and non-process wastewaters are generated from reaction and oxidation scrubbers, spent chemical treatment, product finishing, HCl storage vent scrubber, oxidation unit tank and equipment vents, supernatant from coke and ore solids and wastewater treatment disposal impoundments. Wastewater treatment solids are generated from the neutralization and settling of commingled process and non-process wastewater.

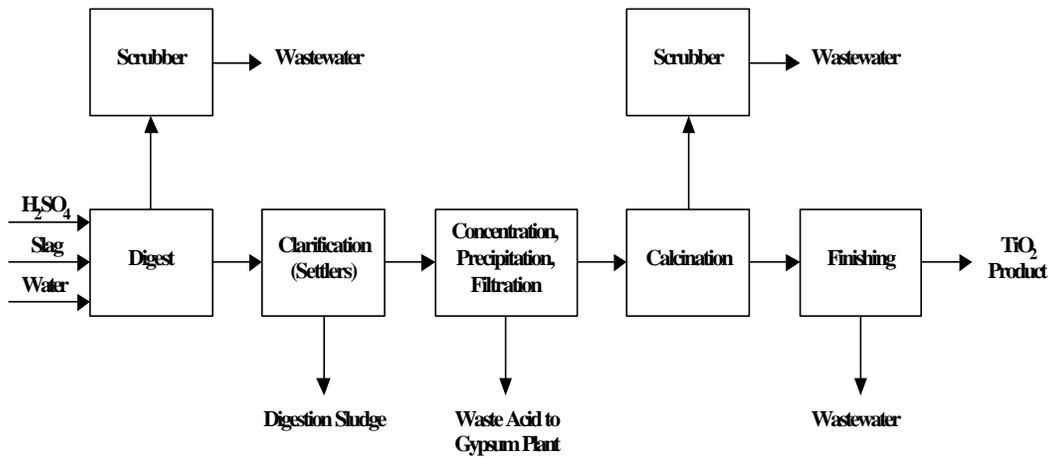
2.2 PRODUCTION TRENDS, CHANGES AND IMPROVEMENTS

The dependence of most of the titanium dioxide producers on Australian rutile, ilmenite, and titaniferrous slags has led to strong price increases for these raw materials over the past years. The U.S. plants that previously produced titanium dioxide by the higher cost sulfate route have been eliminated or updated. In terms of conversion to the chloride process, the U.S. is considerably more advanced than other countries. This advantage will eliminate the capital expenditures associated with the conversion that many other countries will likely be required to make over the next decade in order to remain cost-competitive.¹³

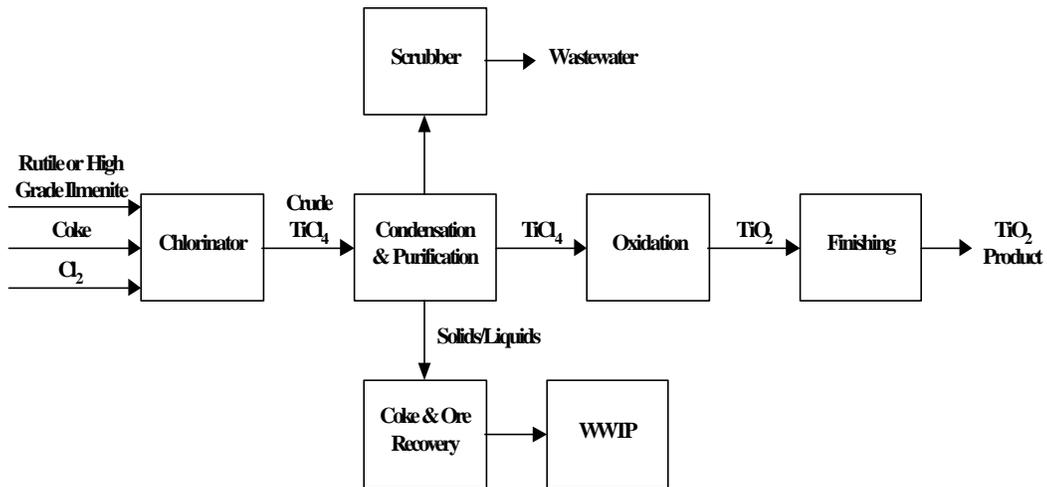
Figure 2.1 - Process Flow Diagram for the Production of Titanium Dioxide

¹³ 54 FR 36592 (Sept. 1, 1989), 55 FR 2322 (Jan. 23, 1990), the July 31, 1990 Report to Congress on Wastes from Mineral Processing, and 56 FR 27300 (June 13, 1991).

SULFATE PROCESS



CHLORIDE/CHLORIDE/ILMENITE PROCESS



3. TITANIUM DIOXIDE WASTE CHARACTERIZATION, GENERATION, MANAGEMENT, SCREENING AND ASSESSMENT

For the purposes of this listing determination, the wastes generated as a result of the production of titanium dioxide via the three production processes were grouped into categories. **Tables 3.1, 3.2 and 3.3** presents a summary of the chloride, sulfate and chloride-ilmenite waste categories that were evaluated as part of this listing determination. **Section 3.1** presents a discussion of the sampling and analysis effort that was conducted to characterize the wastes of concern. **Section 3.2** presents a discussion of the volumes, management practices, and characterization for each of the waste categories presented in Tables 3.1, 3.2 and 3.3. A discussion of the initial risk screening for each waste category is also included as part of the discussion. **Section 3.3** presents a discussion of the waste that are generated on-site at titanium dioxide facilities that are outside the scope of the consent decree.

Table 3.1 - Waste Reported by Titanium Dioxide Facilities Using the Chloride Process

Facility	Commingled Wastewaters from the chloride process	Wastewater Treatment Sludges from commingled chloride and sulfate process wastewaters	Various Sands from milling, scouring and oxidation	Chloride Solids
Kemira Pigments, Inc.	See Table 3.2	x	x	x
Millennium Inorganic Chemicals, Baltimore Plant	See Table 3.2	x	x	x
Kerr-McGee Chemicals Corporation	x		x	x
Kronos/Louisiana Pigment Co.	x			x
Millennium Inorganic Chemicals, Ashtabula Plant I	x			x
Millennium Inorganic Chemicals, Ashtabula Plant II	x			x

Table 3.2 - Waste Reported by Titanium Dioxide Facilities Using the Sulfate Process

Facility	Gypsum from the sulfate process	Digestion Scrubber Water from the sulfate process	Sulfate Process Digestion Sludges	Commingled Wastewaters from chloride and sulfate processes
Kemira Pigments, Inc.	x	x	x	x
Millennium Inorganic Chemicals	x	x	x	x

Table 3.3 - Waste Reported by Titanium Dioxide Facilities Using the Chloride-Ilmenite Process

Facility	Waste Acid (ferric chloride)	Non-Bevill-exempt Nonwastewaters	Commingled wastewaters	Additive vent filters solids	Off-specification TiO₂ Product	Railcar/Trailer Product Wash-out
E.I. DuPont de Nemours DeLisle Plant; Pass Christian, MS	x	x	x	x	x	
E.I. DuPont de Nemours New Johnsonville, TN	x	x	x		x	x
E.I. DuPont de Nemours Edge Moor, DE	x	x	x			

3.1 CHARACTERIZATION OF TITANIUM DIOXIDE WASTES

As part of the information gathering activities, EPA collected and analyzed samples of titanium dioxide production wastes at five facilities: E.I. du Pont de Nemours and Co. in Edge Moor, DE; Kemira Pigments, Inc. in Savannah, GA; E.I. du Pont de Nemours and Co. in New Johnsonville, TN; Kerr-McGee Chemical Corp. in Hamilton, MS; and Millennium Inorganic Chemicals in Baltimore, MD. The sampling and analysis of selected wastes provide the necessary characterization to determine what toxic constituents are present in the wastes and at what concentrations. The waste concentrations in the wastes were used in the risk screening and risk modeling assessments.

Totals, TCLP, and/or SPLP analyses were conducted on each record sample collected for the listing determination. A summary of the analytical results for each sample is presented in Appendix A. The complete set of analytical results, the validation report and a detailed report of the record sampling trip can be found in the following reports:

Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes From the Inorganic Titanium Dioxide Manufacturing Sector; DuPont Edge Moor, DE; September 7, 1999.

Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes From the Inorganic Titanium Dioxide Manufacturing Sector; DuPont New Johnsonville, TN; September 14, 1999.

Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes From the Inorganic Titanium Dioxide Manufacturing Sector; Millennium Inorganics Co., Baltimore, MD; September 23 and September 30, 1999.

Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes From the Inorganic Titanium Dioxide Manufacturing Sector; Kemira Pigments, Co., Savannah, GA; September 9, 1999.

Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes From the Inorganic Sodium Chlorate and Titanium Dioxide Manufacturing Sector for the Kerr-McGee Facility; July 17, 2000

These reports are available in the docket for this rulemaking.

The sampled facilities collected split-samples of some of the samples collected by EPA. The split-sample analytical results for two facilities are found in Appendix B.

3.2 EVALUATION OF TITANIUM DIOXIDE WASTE CATEGORIES

3.2.1 Commingled Wastewaters from the Chloride Process, Including Wastewaters from Coke and Ore Recovery, Scrubber Water, Finishing Wastewaters and Sludge Supernatants

Waste Generation

All six of the facilities that produce titanium dioxide via the chloride process commingle the wastewaters that are generated at various points in the production process. At the two facilities that use the sulfate process, the chloride and sulfate process wastewaters are commingled. The evaluation of these chloride/sulfate wastewaters is discussed in Section 3.2.6 with the “Commingled Wastewaters from the Chloride and Sulfate Process” waste category. The wastewaters generated at the remaining four “chloride only” facilities were assessed as part of this waste category and include:

Wastewater From Coke and Ore Recovery

All four of the “chloride only” facilities generate these wastewaters during the separation of the slurry produced during the initial chlorination reaction. The metal chloride impurities and unreacted coke and ore solids are separated from the titanium tetrachloride produced during the chlorination process.

Scrubber Wastewater (HCl)

Hydrochloric acid (HCl) is generated by all of the “chloride only” facilities as a result of the scrubbing of the off-gas from the chlorination, purification, and oxidation parts of the manufacturing process. These wastewaters are commingled with other wastewaters and treated in each facility’s waste water treatment system and are addressed in this category.

In addition, three of the chloride process facilities also report reusing a portion of the scrubber waters as hydrochloric acid. Millennium Plant I in Ohio uses the scrubber water onsite in titanium dioxide production and sells it as HCl for steel pickling. Kerr-McGee sends a portion of the scrubber water as HCl to their sister facility in Mobil, GA to be used in beneficiation (leaching) of ilmenite ore. LA Pigments uses a portion of the scrubber water in the titanium dioxide process and sells a portion of their scrubber water as HCl to be used as an acidizing agent in the oil field industry. According to the facility, this HCl meets all the required specifications for HCl.

Finishing Wastewaters

Finishing wastewaters are generated in the product finishing operation. The wastewater is commingled with other process wastewaters for treatment prior to NPDES discharge.

Sludge Supernatant

Both of the Millennium facilities in Ohio generate wastewaters as a result of the filtering of the

sludge from the surface impoundments that are a part of the facility’s wastewater treatment systems. The supernatant is recycled to the headworks of the wastewater treatment system for treatment.

Waste Management Practices

Three of the four of the “chloride only” facilities commingle these wastewaters in on-site wastewater treatment systems that are comprised of tanks and surface impoundments. Kronos/Louisiana Pigments Co. uses an entirely tank-based treatment system.

Kerr-McGee Chemical Corporation

At this facility, the wastewater treatment system consists of tanks, which are used to neutralize the wastewater (and commingled Bevill-exempt solids), and a series of three impoundments for settling. The treated wastewater from the tank portion of the system is sent to the settling ponds. The first two impoundments are lined and the last is unlined. The treated wastewaters are discharged via an NPDES permit, with the settled solids remaining in the surface impoundments. The facility plans to close the impoundments in place when the sludge storage capacity is reached.

Kronos/Louisiana Pigment Company

This facility uses a tank-based system to neutralize their commingled wastewaters. Although the facility uses surface impoundments onsite for managing other wastewaters such as stormwaters, they do not use the surface impoundments to manage the wastewaters from the titanium dioxide manufacturing process. The treated wastewaters are discharged via an NPDES permit.

Millennium Inorganic Chemicals Plants I and II; Ashtabula, Ohio

At these two facilities, the wastewater treatment systems are comprised of a tank and surface impoundments. The commingled wastewaters are neutralized in a tank and settled in the surface impoundments. The treated wastewaters are discharged via NPDES permits.

The management of these commingled wastewaters in surface impoundments prior to discharge at Kerr-McGee and the Millennium Ashtabula facilities was evaluated for potential risks to human health and the environment via groundwater releases to drinking water wells and surface water. **Table 3.4** presents of all the wastewaters, with their associated volumes, that are managed in the wastewater treatment systems at each facility.

Table 3.4 - Commingled Wastewaters from the Chloride Process		
Facility	Wastewater (RIN #)	Total Volume (MT/yr)
Kerr-McGee Chemical Corporation	Wastewater from Coke and Ore Recovery (RIN 1)	7,356,798

Table 3.4 - Commingled Wastewaters from the Chloride Process		
Facility	Wastewater (RIN #)	Total Volume (MT/yr)
Kronos/Louisiana Pigment Company	Wastewater from Coke and Ore Recovery (RIN 4)	63,394
	Scrubber Wastewater (RIN 2)	5,186
Millennium Inorganic Chemicals, Plant I; Ashtabula, OH	Wastewater from Coke and Ore Recovery (RIN 1)	70,000
	Scrubber Wastewater (RIN 2)	13,900
Millennium Inorganic Chemical Plants II; Ashtabula, Ohio	Wastewater from Coke and Ore Recovery (RIN 1)	90,000
	Scrubber Wastewater (RIN 2)	13,900

Waste Characterization

The commingled wastewaters at Kerr-McGee are representative of the four “chloride only” facilities and was selected for sampling and analysis. The Sample KM-SI-01 collected during record sampling was used to characterize this waste category. The sample was collected at the inlet to the surface impoundment train.¹⁴ This waste contained solids and waste acids from the chlorination process that had been mixed with other wastewaters. To isolate the impact of the wastewater on the environment from that of the sludge, the analyses conducted on the sample were as follows: matrix (totals, TCLP and SPLP), filtrate SPLP, and leachate SPLP. The SPLP filtrate portion of the sample is assumed to be representative of the wastewater throughout the settling ponds and was used to conduct an initial screening analysis and subsequent full risk assessment modeling.

Table 3.5 presents the analytical results for the constituents detected in the filtrate with the corresponding the Health-Based Levels (HBLs) and/or Ambient Water Quality Criteria (AWQC). For details on the HBLs and AWQC please refer to *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)*.

Results of Initial Screening Analysis

EPA determined that the surface impoundment scenarios (Kerr-McGee and Millennium facilities) pose a more significant potential risk than the tank based scenario (Louisiana Pigment) because it was assumed that wastewater treatment tanks retain sufficient structural integrity to prevent wastewater releases to the subsurface (and therefore to groundwater). Also, overflow and spill controls prevent significant wastewater releases. The impoundments at the three “chloride only” facilities were considered separately because there was no indication that the waste would be

¹⁴This facility also commingles wastewaters from sodium chlorate production, which accounts for a small percentage (<7%) of the total waste volume.

managed in any units other than those reported.

The surface impoundment at Kerr-McGee required further assessment beyond the initial screening analysis for infiltration to the river pathway and the groundwater contamination pathway due to exceedences of both the HBL and AWQC.

Millennium Ashtabula, Ohio Facilities

At these two facilities, the impoundments that make up the wastewater treatment system are located on or near the Fields Brook, which feeds into the Ashtabula River, that ultimately feeds into Lake Erie (2 miles away). See map in *Fields Brook Project, SCM Plant 2, TiO₂ Facility, Phase I RI Report, Rev. 1*. The localized groundwater flow is south toward the creeks, and the deeper flow is northward to Lake Erie. The facility was not able to identify any private drinking water wells in the vicinity of the plant. All land between the facility and the Lake is industrial. A Superfund multimedia study of this area¹⁵ indicated that the groundwater is in a very low permeability formation, and that the public drinking water supply is from the lake. No further consideration of this scenario was required since the potential exposure via the drinking water well scenario was assessed at the Kerr-McGee facility.

Kerr-McGee

The SPLP filtrate results for Sample KM-SI-01 were used to screen any surface water releases and possible drinking water well contamination resulting from the management of this waste in the final surface impoundment of the facility's wastewater treatment system.

The surface water release pathway for the wastewater from the settling ponds is to the nearby Tombigbee River. The *RCRA Facility Assessment (RFA) of Kerr-McGee Chemical Corporation; Hamilton, MS; June 16, 1995; pp. II-44-45* states that "the groundwater flow near the surface impoundments is to the northwest and discharges into on-site swamps. Regionally, however, the groundwater flow direction is to the southwest and discharges into the Dose Maie Creek and the Tombigbee River." Kerr-McGee owns all of the land between the impoundments and the river (including the creek), which appears to be swampy and undeveloped on available maps. The potential infiltration of wastewater from the final unlined surface impoundment into the river were assessed for risk.

The drinking water release pathway for the wastewater from the impoundment is to potential drinking water wells in the area. The RFA states "The Kerr-McGee facility is located approximately one mile southwest of New Hamilton, MS and two miles from the Sulfur Springs School. According to a 1991 EPA Chemical Safety Audit, the site is located in a predominantly agricultural setting. EPA estimates that there are less than 3,000 people living within 6 miles of the plant; however, some of the residents own property adjacent to Kerr-McGee. At least six off-

¹⁵Fields Brook Project, SCM Plant 2, TiO₂ Facility, Phase I RI Report, Rev. 1, 8/24/94.

site ground-water wells are located in close proximity to the northern boundary of the site.¹⁶ The Hamilton facility encompasses wetland areas along the western and southern portions of the site and along McKinley Creek.” See *1998 RCRA 3007 Survey of Inorganic Chemicals Industry* for Kerr-McGee Chemical for applicable maps. Based on USGS data obtained from the state, a residential well (Q050) was reported just off the property boundary at 5,000 feet from the impoundment of concern. The closest property boundary to this impoundment is 2,000 feet. Due to uncertainty in groundwater flow direction (localized flow to the northwest in the vicinity of the impoundments, overall flow to the southwest), the potential impact on potential drinking water wells to the north was assessed for risk.

The constituents of concern that were detected above the HBLs in Kerr-McGee’s wastewater are antimony, arsenic, molybdenum and thallium. The constituents of concern that exceeded AWQC are antimony, arsenic, thallium and manganese.

The drinking water release pathway for the wastewater from the impoundment is to potential drinking water wells in the area. The RFA states “The Kerr-McGee facility is located approximately one mile southwest of New Hamilton, MS and two miles from the Sulfur Springs School. According to a 1991 EPA Chemical Safety Audit, the site is located in a predominantly agricultural setting. EPA estimates that there are less than 3,000 people living within 6 miles of the plant; however, some of the residents own property adjacent to Kerr-McGee. At least six off-site ground-water wells are located in close proximity to the northern boundary of the site.¹⁷ The Hamilton facility encompasses wetland areas along the western and southern portions of the site and along McKinley Creek.” See *1998 RCRA 3007 Survey of Inorganic Chemicals Industry* for Kerr-McGee Chemical for applicable maps. Based on USGS data obtained from the state, a residential well (Q050) was reported just off the property boundary at 5,000 feet from the impoundment of concern. The closest property boundary to this impoundment is 2,000 feet. Due to uncertainty in groundwater flow direction (localized flow to the northwest in the vicinity of the impoundments, overall flow to the southwest), the potential impact on potential drinking water wells to the north was assessed for risk.

Constituent	KM-SI-01 SPLP filtrate (mg/L)	HBL (mg/L)	AWQC (mg/L) (freshwater/saltwater)
Aluminum	0.013	16	0.087
Antimony	0.044	0.0063	0.014

¹⁶Phone log. Ron Josephson (EPA) to Mr. Jim Hoffman, Mississippi Department of Environmental Quality, Office of Land and Water Resources. December 22, 1999.

¹⁷Phone log. Ron Josephson (EPA) to Mr. Jim Hoffman, Mississippi Department of Environmental Quality, Office of Land and Water Resources. December 22, 1999.

Table 3.5 - Initial Screening Analysis for Commingled Wastewaters from Chloride Process

Constituent	KM-SI-01 SPLP filtrate (mg/L)	HBL (mg/L)	AWQC (mg/L) (freshwater/saltwater)
Arsenic	0.001 *	0.00074	0.000018
Barium	0.23	1.1	NA
Beryllium	<0.002	0.031	NA
Boron	0.39	1.4	NA
Cadmium	<0.005	0.0078	0.0022/0.0093
Calcium	2,940	NA	NA
Chromium	<0.005	23	0.74
Cobalt	<0.005	0.94	NA
Copper	0.007	1.3	0.0090/0.0031
Iron	<0.05	5	1
Lead	<0.003	0.015	0.0025/0.0081
Magnesium	60.5	NA	NA
Manganese	0.46	0.73	0.05
Mercury	<0.0002	0.0047	0.000050
Molybdenum	0.23	0.078	NA
Nickel	<0.005	0.31	0.052/0.0082
Potassium	18.6	NA	NA
Silver	<0.001	0.078	0.0034/0.0019
Sodium	606	NA	NA
Thallium	<0.005 ¹⁸	0.0013	0.0017
Tin	<0.01	9.4	NA
Titanium	<0.005	NA	NA
Vanadium	0.008	0.14	NA
Zinc	<0.05	4.7	0.12 /0.081

¹⁸Thallium is identified as a potential constituent of concern because (1) it was detected in the totals analysis (0.086 mg/L) at levels exceeding the HBL and AWQC, and (2) the SPLP filtrate analysis detection limit was too high to confirm that mobile levels of thallium do not exceed these standards.

Table 3.5 - Initial Screening Analysis for Commingled Wastewaters from Chloride Process

Constituent	KM-SI-01 SPLP filtrate (mg/L)	HBL (mg/L)	AWQC (mg/L) (freshwater/saltwater)
Dioxins/Furans, (ng/L)			
2378-TCDF (TEF=0.1) ¹⁹	<0.11	NA	NA
Total TCDF	<0.11	NA	NA
2378-TCDD	<0.11	NA	NA
Total TCDD	<0.11	NA	NA
12378-PeCDF (0.05)	<0.56	NA	NA
23478-PeCDF (0.5)	<0.56	NA	NA
Total PeCDF	<0.56	NA	NA
12378-PeCDD (TEF=1)	<0.56	NA	NA
Total PeCDD	<0.56	NA	NA
123478-HxCDF (0.1)	<0.56	NA	NA
123678-HxCDF (0.1)	<0.56	NA	NA
234678-HxCDF (0.1)	<0.56	NA	NA
123789-HxCDF (0.1)	<0.56	NA	NA
Total HxCDF	<0.56	NA	NA
123478-HxCDD (0.1)	<0.56	NA	NA
123678-HxCDD (0.1)	<0.56	NA	NA
123789-HxCDD (0.1)	<0.56	NA	NA
Total HxCDD	<0.56	NA	NA
1234678-HpCDF (0.01)	<13	NA	NA
1234789-HpCDF (0.01)	<13	NA	NA
Total HpCDF	<13	NA	NA
1234678-HpCDD (0.01)	<13	NA	NA
Total HpCDD	<13	NA	NA

¹⁹TEF= Toxicity Equivalent Factor, provided in parentheses after congener name. Dioxin TEQs calculated using WHO-TEFs.

Constituent	KM-SI-01 SPLP filtrate (mg/L)	HBL (mg/L)	AWQC (mg/L) (freshwater/saltwater)
OCDF (0.0001)	<1.1	NA	NA
OCDD (0.0001)	<1.1	NA	NA
2378-TCDD TEQ	0 ng/L	0.0071 ng/L	0.0031 ng/L

NA- Not Available

NR- Not Reported In Analytical Data Report

*Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.

For the Commingled Wastewaters from Chloride Process waste category, the potential groundwater releases to both surface water and drinking wells were assessed as described above. The air pathway was not assessed for this waste category because no volatile organic or other constituents that pose risk due to air releases were detected. Please refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the risk assessment.

3.2.2 Various Sands from Oxidation, Milling, and Scouring

Waste Generation and Management

Kemira Pigments and Millennium Inorganic Chemicals in Baltimore reported generating milling sands during finishing operations. Kemira Pigments also generates a scouring sand during product finishing. Kerr-McGee reported generating an “oxidation sand” when silica sand is used to remove the crystallized titanium dioxide from cooling tub surfaces after the oxidation step of the chloride process.

Kemira Pigments sends their waste sand to an off-site Subtitle D landfill. Millennium Inorganic Chemicals (Baltimore, MD) sends their waste sand to an on-site landfill. Kerr-McGee sends this waste to an on-site dedicated industrial Subtitle D landfill. **Table 3.6** presents the chloride process waste sands, with the associated volumes and management practices, reported by each facility.

Facility	Waste Sand (RIN #)	Management	Total Volume (MT/yr)
Kemira Pigments	Milling Sand (RIN 10)	Off-site industrial Subtitle D landfill	200

Milling, and Scouring

Facility	Waste Sand (RIN #)	Management	Total Volume (MT/yr)
	Scouring Sand (RIN 8)	Off-site industrial Subtitle D landfill	2,300
Millennium Inorganic Chemicals; Baltimore, MD	Milling Sand (RIN 14)	On-site Subtitle D landfill	50
Kerr-McGee	Oxidation Sand (RIN 2)	On-site Subtitle D monofill	6,935

Waste Characterization

During record sampling, two samples were collected from the Kemira Pigments facility to characterize this waste category. Sample KP-SO-04 of the scouring sand was taken from a container holding this material by compositing four grab samples of the material; a milling sand sample (KP-SO-05) was collected by compositing four grab samples of this wet slurry-like material from a container holding this material. Although a sample of oxidation sand was not available during the record sampling time frame, the sand is assumed to be similar in composition to the milling and scouring sands because they are all associated with product finishing operations.

Results of Initial Screening

Milling Sand

Since the milling sand is managed in an industrial Subtitle D landfill, the SPLP results for Sample KP-SO-05 were compared against the HBLs for each constituent to determine if further risk assessment was necessary. **Table 3.7** presents the analytical results for the constituents detected in the SPLP and the corresponding HBLs and/or AWQC. The only constituent detected above the HBL was antimony. Therefore, further analysis of the risk for antimony under the industrial Subtitle D landfill scenario was assessed for the groundwater ingestion pathway. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the risk assessment.

Constituent	KP-SO-05 SPLP (mg/L)	HBL (mg/L)
Aluminum	<0.1	16
Antimony	0.024	0.0063
Barium	0.21	1.1
Boron	0.19	1.4
Calcium	0.73	NA
Chromium	<0.005	23
Chromium, +6	<0.02**	0.047

Table 3.7 - Initial Screening Analysis for Milling Sand		
Constituent	KP-SO-05 SPLP (mg/L)	HBL (mg/L)
Copper	0.003	1.3
Iron	<0.05	5
Magnesium	0.083	NA
Manganese	<0.005	0.73
Nickel	0.044	0.31
Sodium	4.0	NA
Tin	<0.01	9.4
Titanium	0.12	NA
Zinc	0.032*	4.7

*Results are less than the typical laboratory reporting limit, but are greater than calculated instrument DL.

**Determined from DI leach.

NA- Not Available

Scouring Sand

The relevant risk scenarios for the scouring sand waste are an on-site Subtitle D industrial landfill (monofill) at Kerr-McGee and an off-site Subtitle D industrial landfill at Kemira. The SPLP results for KP-SO-04 are believed to be representative of the leachate from both landfill scenarios. **Table 3.8** presents a comparison of the analytical results for the constituents detected in the SPLP leachate and the corresponding HBLs and AWQC. The antimony concentration in the SPLP leachate exceeds the antimony the HBL only by a factor of 1.1. Since direct ingestion of the off-site landfill leachate is highly unlikely, it is assumed that antimony does not pose a risk via groundwater ingestion under either on-site or off-site industrial landfill scenarios.

However, aluminum and mercury were detected above the AWQC by a factors of 3 and 8 (respectively) and are constituents of concern for the on-site landfill scenario via the surface water pathway. Subsurface releases from this landfill may reach the Tombigbee River, approximately 500 feet to the west. The Kerr-McGee RFA states that “the groundwater flow near the surface impoundments (the landfill is on the southern side of the impoundments) is to the northwest and discharges into onsite swamps. Regionally, however, the groundwater flow direction is to the southwest and discharges into the Dose Maie Creek and the Tombigbee River.” It is highly likely that the 500' subsurface transport scenario and the dilution into the river scenario would result in a DAF significantly greater than eight thus bringing the mercury and aluminum concentration below the AWQC. Therefore, it is assumed this waste screens out. Further support to this assumption is the fact that the landfill has a double liner and a leachate collection system.

Note that unlike the surface impoundments that manage “commingled chloride wastewaters” and that were assessed for potential releases to drinking water wells at this site, the sand landfill is located at the southeast corner of Kerr-McGee’s property approximately 1800 feet (center-to-center) to the southwest of the modeled surface impoundment. Groundwater flows in the vicinity of this landfill are unlikely to move toward the wells assessed for the surface impoundment.

Table 3.8 - Initial Screening Analysis for Scouring Sand

Constituent	KP-SO-04 SPLP (mg/L)	HLB (mg/L)	AWQC (mg/L)
Aluminum	0.23	16	0.087
Antimony	0.007	0.0063	0.014
Barium	0.11	1.1	1
Boron	0.15	1.4	NA
Calcium	0.96	NA	NA
Chromium	0.018	23	0.74
Copper	0.004	1.3	0.0090
Iron	<0.05	5	1
Lead	0.001*	0.015	0.0025
Magnesium	0.066	NA	NA
Manganese	0.006	0.73	0.05
Mercury	0.0004	0.0047	0.000050
Nickel	0.019	0.31	0.052
Sodium	8.4	NA	NA
Tin	<0.01	9.4	NA
Titanium	0.068	NA	Na
Zinc	0.067	4.7	0.12

*Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.

NA- Not Available

3.2.3 Gypsum from the Sulfate Process

Waste Generation and Management

Gypsum is produced from the sulfate process when the waste acid generated from the filtering of

the titanium dioxide hydrate solution is neutralized with calcium carbonate (CaCO₃). At the Millennium Baltimore facility, a secondary gypsum is produced when the filtrate from the initial neutralization is sent through a secondary neutralization process. During the secondary neutralization step, more CaCO₃ is added and the slurry is mixed and filtered. At both Kemira and Millennium, the treated wastewater formed during the neutralization process is discharged via permitted NPDES outfalls.

At both facilities the gypsum is stored in piles for drying (without pads or liners) before it is sold for commercial use. Kemira sells its gypsum for use in the manufacture of wallboard, cement, agricultural chemicals or chemical products. At Millennium, primary gypsum is sold for use in wallboard or sent to the facility's on-site Subtitle D landfill. The secondary gypsum also is sent to the on-site landfill. **Table 3.9** provides the management practices and volumes for the gypsum generated at both facilities.

Facility	Waste (RIN #)	Management Practice	Total Volume (MT/yr)
Millennium Inorganics; Baltimore, MD	Primary Gypsum (RIN 10)	Storage in piles, sold for use in wallboard	160,027
	Primary Gypsum* (RIN 10)	Storage in piles, sent to facility's off-site landfill	17,781
	Secondary Gypsum (RIN 12)	Storage in piles, sent to facility's off-site landfill	51,710
Kemira Pigments; Savannah, GA	Primary Gypsum (no RIN assigned)	Storage in pile, sold for use in agricultural chemicals (use constituting disposal)	Not Reported
		Storage in pile, sold for use in cement	Not Reported
		Storage in pile, sold for use in chemical products	Not Reported
		Storage in pile, sold for use in wallboard	Not Reported

*The facility reported during a site visit that about 10% of the primary gypsum produced is sent to the landfill, and the rest is sold for use in wallboard.

Waste Characterization

Samples of the primary and secondary gypsum were collected during record sampling to characterize this waste. At Kemira Pigments Inc., a sample (KP-SO-O1) of the primary gypsum was collected by compositing four scoops of this wastestream from the perimeter of a pile of this material discharged by a conveyor belt directly from the process. At Millennium Inorganics in

Baltimore, MD, samples of both the primary (MI-SO-04) and the secondary (MI-SO-03) gypsum were collected by compositing scoops of the material from separate locations around the perimeter of similarly-generated piles of gypsum.

Results of Initial Screening

The initial screening of this waste category considered each of the reported management scenarios that involve land placement: agricultural chemicals, cement, piles and landfills. The potential releases to both air and groundwater were evaluated.

Table 3.10 presents the analytical results for the constituents detected in the relevant samples with the corresponding applicable regulatory limit (HBLs, AWQC, etc.).

Table 3.10 - Initial Screening Analysis for Primary and Secondary Gypsum

Constituent	Primary gypsum (KP-SO-01)		Primary gypsum (MI-SO-04)		Secondary gypsum (MI-SO-03)		HBL (mg/L)	AWQC (mg/L)	SSL (1) (mg/kg)
	Total (mg/kg)	SPLP (mg/L)	Total (mg/kg)	SPLP (mg/L)	Total (mg/Kg)	SPLP (mg/L)			
Aluminum	2,210	<0.011 (1)	227	0.24	6,420	<0.1	16	0.087	47,000
Antimony	0.6	0.02	<0.5	0.014	3.2	0.055	0.0063	0.014	32*
Arsenic	<0.5	<0.005	<0.5	<0.005	0.8	<0.0035	0.00074	0.000018	5
Barium	0.8	0.03	1.2	0.02 (2)	7.9	0.033	1.1	1	440
Boron	<10	0.10	<5	0.06	6.1	0.28	1.4	NA	26
Cadmium	<0.5	<0.005	<0.5	<0.005	1.7	<0.005	0.0078	0.0022	4
Calcium	135,000	648	189,000	634	135,000	662	NA	NA	NA
Chromium	232	<0.005	30.5	<0.005	693	0.001 (2)	23	0.74	120,000*
Cr,+6	<0.40	<0.02 (3)	<0.40	<0.02 (3)	32.7	<0.02 (3)	0.047	0.011	37
Cobalt	<0.5	<0.005	<0.5	<0.005	3.9	<0.005	0.94	NA	6.7
Copper	0.9	0.003	<0.5	0.003	2.4	0.005	1.3	0.0031	17
Iron	953	<0.05	767	<0.05	59,000	<0.05	5	1	430,000*
Lead	1.5	0.002 (2)	<0.5	0.002 (2)	<0.5	0.003 (2)	0.015	0.0025	16
Magnesium	224	9.6	121	5.85	896	33.7	NA	NA	NA
Manganese	13.3	0.13	9.3	0.13	673	3.1	0.73	0.05	3,800
Molybdenum	<0.5	<0.005	<0.5	<0.005	0.9	<0.005	0.078	NA	400*
Nickel	<0.5	<0.005	<0.5	<0.005	10.5	0.009	0.31	0.0082	13

Table 3.10 - Initial Screening Analysis for Primary and Secondary Gypsum

Constituent	Primary gypsum (KP-SO-01)		Primary gypsum (MI-SO-04)		Secondary gypsum (MI-SO-03)		HBL (mg/L)	AWQC (mg/L)	SSL (1) (mg/kg)
	Total (mg/kg)	SPLP (mg/L)	Total (mg/kg)	SPLP (mg/L)	Total (mg/Kg)	SPLP (mg/L)			
Selenium	<0.5	<0.005	<0.5	<0.005	0.7	<0.005	0.078	0.0050	400*
Sodium	108	5.9	135	3.3	2,260	10	NA	NA	NA
Thallium	2.9	<0.0022	<2	<0.005	3.5	0.0006	0.001	0.0017	6*
Tin	2.7	<0.01	<1	<0.01	2.9	<0.01	9.4	NA	48,000*
Titanium	5,430	<0.005	377	<0.005	5,900	<0.005	NA	NA	NA
Vanadium	641	0.027	62.2	0.027	1,930	0.014	0.14	NA	4,000#
Zinc	21.9	0.033 (2)	<5	0.026 (2)	<5	0.040 (2)	4.7	0.081	48

(1) This involves a three tiered screen: a. background soils; b. soil ingestion HBL (*); and c. air characteristic level (#). The vanadium soil ingestion level is 720 mg/kg; the air characteristic level represents a distance of 25 m for waste piles.

(2) Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.

(3) Determined from DI leach

(4) Salt water AWQC

NA- Not Available; ND- Not Detected

Cement and Agricultural Use Scenarios

Kemira uses on-specification gypsum in cement. This cement scenario was screened out by comparing the total levels for Sample KP-SO-01 to Soil Screening Levels (SSL). The agricultural chemicals scenario is associated with Kemira which sells the gypsum to peanut farmers for use as a nutrient used to harden the peanut shells. This scenario also was screened by comparing the total constituent analyses to the Soil Screening Levels. All constituents are below these levels and thus this scenario screens out.

Landfill Scenario

As discussed previously, the gypsum is landfilled at the Millennium facility in Baltimore. In addition, CPC (a barium carbonate manufacturer) indicated that they purchase red gypsum from Kemira for use in treating their wastes. CPC's wastes are subsequently landfilled. The assessment of the landfill scenario is discussed below.

The Millennium Inorganic Chemicals (Baltimore, MD) landfill was screened for impacts via the groundwater pathway from the primary and secondary gypsum since both are placed in the landfill. The primary and secondary gypsum were assessed separately because they are generated at different places in the process. The SPLP results for both the primary and the secondary gypsum were used to screen potential releases to groundwater since there is no contact with municipal landfill leachate in the reported management practices. For the primary gypsum (MI-SO-04), antimony was detected above the HBL, and aluminum and manganese were detected above the AWQC. For the secondary gypsum (MI-SO-03), the constituents detected above the HBL were antimony, manganese, and arsenic. The constituents detected above the AWQC are manganese and arsenic. Copper and nickel were detected above the saltwater AWQC.

The primary gypsum contained lower levels of leachable metals than the secondary gypsum so the assessment focused on the secondary gypsum as it was more likely to show risk and the management scenarios are identical (they are placed in the same on-site industrial landfill). Furthermore, the volume of the primary gypsum sent to the landfill was smaller. Therefore, the on-site Subtitle D industrial landfill scenario was assessed for secondary gypsum via the surface water pathway and potential drinking water pathway because of the HBL and AWQC exceedences. (See Section 3.2.5 for a discussion of the selection of these modeling pathways - this onsite landfill was modeled for several wastes.) See 1998 RCRA 3007 Survey for the Inorganic Chemicals Industry for the Millennium facility in Baltimore, MD for the relevant maps showing unit locations. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the risk assessment.

Piles Scenario

As discussed above, both facilities use piles to store and dry their gypsum prior to landfilling or sales. We believe neither site uses pads or liners for these piles and so the groundwater and air pathway associated with the pile scenario were assessed. For Millennium, the landfill scenario for this waste, which is more conservative due to its size in comparison to the pile, is being assessed. Therefore, no further assessment of the groundwater impact from the piles at this site is

necessary.

For Kemira, the SPLP leachate results (because the waste is managed onsite with no potential contact with municipal landfill leachate) for Sample KP-SO-01 were used to screen the groundwater pathway associated with the pile scenario. Antimony was detected above the HBL and manganese was detected above the AWQC; both exceedences were minor. Initially, the gypsum is placed on “gypsum hills” for two weeks for drying, and then moved to piles under a roof (no side walls) prior to sales. The risk assessment was not conducted on the potential impact of drinking water wells because a risk assessment for the more conservative Millennium landfill scenario was conducted. Kemira is unaware of any drinking water wells in the vicinity and the pile is substantially smaller than the Millennium landfill. The Kemira waste also contains lower toxic constituent levels than Millennium. EPA assumed the Kemira surface water scenario screens out based on the (a) low required dilution attenuation factor (DAF) to reduce exposure concentrations below HBLs, (b) small exposed pile surface area, estimated dimensions of 30 feet in diameter and 12 feet in height, (c) the 3,500 foot distance to the two nearby rivers, and (d) expected large dilution in either of the two rivers. See USGS map in Appendix C for map of facility and adjoining water bodies.

For the air pathway, both facilities place their piles outside in exposed areas. This scenario was assessed by comparing all constituent levels to soil screening levels. In all cases the constituents were below these levels. All were below the direct soil ingestion levels, except for one sample of vanadium in secondary gypsum (this makes up a small fraction of the gypsum generated at the site), which was only 2.7 times the ingestion level. It is highly unlikely that any particulate release from the waste pile would approach the soil ingestion level for this constituent. Furthermore, the vanadium level is below the air characteristic level, which assessed risks from direct inhalation.²⁰ Therefore, air releases from the pile were not assessed further.

3.2.4 Digestion Scrubber Water from the Sulfate Process

Waste Generation Management

Digestion scrubber wastewaters are generated when the vented gases from the digestion process are scrubbed to remove the acidic components. Both facilities that produce titanium dioxide via the sulfate process generate this waste. At Millennium Inorganic Chemicals (Baltimore, MD), this waste is neutralized and sent to a dedicated settling pond. The neutralized wastewater is discharged via an NPDES permitted outfall. At Kemira Pigments, Inc., the sulfate process digestion scrubber water is commingled with wastewaters from the chloride and sulfate process in the facility’s wastewater treatment system. Kemira’s sulfate process digestion scrubber wastewater is assessed as part of the “Commingled Wastewaters from the Chloride and Sulfate Processes” waste category in Section 3.2.6. **Table 3.11** presents the management practices and the volumes of the digestion scrubber water from the sulfate process at each facility.

²⁰U.S. Environmental Protection Agency, "Revised Risk Assessment for the Air Characteristic Study", EPA 530-R-99-019a, November 1999, Table 4-3.

Table 3.11 - Waste Management Practices and Volumes for Digestion Scrubber Water from the Sulfate Process		
Facility (RIN #)	Management	Total Volume (MT/yr)
Kemira Pigments, Inc. (RIN 1)	Tank, surface impoundment	298,000
Millennium Inorganic Chemicals; Baltimore, MD (RIN 5)	Dedicated surface impoundment	1,702,333

Waste Characterization

A sample was collected from Millennium Inorganic Chemicals to characterize the sulfate process scrubber wastewater. Sample MI-WW-03 was collected from a pipe that transports the wastewater into the settling pond. Total analyses were conducted on the sample collected. TCLP and SPLP analyses were not necessary because the solids content was within the method criterion.

Results of Initial Screening

This surface impoundment scenario was screened using the analytical results for Sample MI-WW-03. The surface impoundment is separated from the Patapsco River by a dike. In addition, Maryland DEQ made the facility install an asphaltic slurry wall between the impoundment and the river. There are recovery wells at the slurry wall that collect groundwater, which is then sent to the wastewater treatment system for processing. Groundwater flow is east towards the river. **Table 3.12** presents the analytical results for the constituents detected in the filtrate with the corresponding HBLs and/or AWQC. The constituents detected²¹ above the AWQC were aluminum, manganese, and mercury. Copper was detected above the saltwater AWQC.

This dedicated surface impoundment was assessed for potential surface water releases. The drinking water well scenario is not of concern since there are no HBL exceedences of concern. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the full risk assessment.

Table 3.12 - Initial Screening Analysis for Digestion Scrubber Water from the Sulfate Process			
Constituent	MI-WW-03 Total (mg/L)	HBL (mg/L)	AWQC (mg/L)
Aluminum	0.58	16	0.087
Antimony	0.010 B	0.0063	0.014

²¹Antimony also exceeded the HBL (1.7xHBL), but at such low levels that it was assumed it would screen out. Antimony also was detected in the equipment blank at 0.05 mg/L.

Table 3.12 - Initial Screening Analysis for Digestion Scrubber Water from the Sulfate Process			
Constituent	MI-WW-03 Total (mg/L)	HBL (mg/L)	AWQC (mg/L)
Barium	0.041	1.1	1
Boron	1.35	1.4	NA
Chromium	0.013	23	0.74
Chromium, hexavalent	<0.02	0.047	0.011
Copper	0.006	1.3	0.0031
Iron	1.53	5	1
Manganese	0.58	0.73	0.05
Mercury	0.0032	0.0047	0.000050
Molybdenum	0.006	0.078	NA
Nickel	0.008	0.31	0.0082
Titanium	0.44 B	NA	NA
Vanadium	0.03	0.14	NA

NA- Not available

3.2.5 Sulfate Process Digestion Sludge

Waste Generation and Management

This sludge is generated as a result of the clarification of the titanyl sulfate liquor that is produced during the digester step. Both of the facilities that produce titanium dioxide via the sulfate process reported generating this waste. Dedicated management units are used at both facilities to manage this waste.

Kemira Pigments Inc, reported generating 34,000 metric tons per year of this waste (RIN 2) which is neutralized in a tank in the first step of the waste treatment process. The settled solids from the neutralization tank (RIN 13) are then sent to a dedicated surface impoundment where the solids are settled out. The solids remain in place and the supernatant effluent from this pond is sent to the wastewater treatment system and is commingled with other process wastewaters from the sulfate and the chloride process in the wastewater treatment system. At Millennium Inorganic Chemicals, this waste is generated during clarification of the titanyl sulfate product to remove unreacted solids and other impurities. The solids are filtered out using a filter press, accumulated in temporary waste piles, and then sent to the facility's on-site Subtitle D industrial landfill via dump truck. **Table 3.13** presents the waste management practices and volumes for the digestion sludge at both facilities.

Table 3.13 - Waste Management Practices and Volumes for Sulfate Process Digestion Sludge		
Facility (RIN #)	Management Practice	Total Volume (MT/yr)
Kemira Pigments, Inc. (RIN 13)	Solids settling in dedicated unlined surface impoundment, supernatant wastewater goes to wastewater treatment system	17,000
Millennium Inorganic Chemicals; Baltimore, MD (RIN 7)	On-site Subtitle D industrial landfill	24,494

Waste Characterization

Two samples of this waste were collected during record sampling for characterization purposes, one at each of the generating facilities.

At Kemira Pigments, Inc. Sample KP-SO-03 was collected from a small weir at the point of neutralization prior to the neutralized slurry going to the dedicated surface impoundment. It was not practical to collect a sample of the sludge from the impoundment due to limited accessibility. At the Millennium Baltimore facility, Sample MI-SO-02 was collected directly after the filter press by compositing four scoops of the solids from four locations around the covered waste pile.

Totals, TCLP, and SPLP analyses were conducted on the samples. A summary of the analytical results for each sample is presented in Appendix A. Detailed reports of the record sampling trip, the complete set of analytical data and the validation reports are available in the “Sampling and Analytical Data Report For Record Sampling and Characterization of Wastes from the Inorganic Titanium Dioxide Manufacturing Sector” for Millennium Inorganic Chemicals (Baltimore, MD) and Kemira Pigments. These reports are available in the docket for this rulemaking.

Results of Screening Analysis

Summary

The on-site landfill at Millennium required further assessment for (1) infiltration and dilution of leachate into the Patapsco River and (2) landfill leachate contamination of potential drinking water wells in the vicinity. The surface impoundment at Kemira did not require further assessment based on initial screening against health based criteria.

Both scenarios, the on-site landfill and surface impoundment, were screened by comparing actual SPLP leachate analytical results with the HBL and AWQC for each constituent. **Table 3.14** presents the analytical results for the constituents detected in the SPLP leachate with the corresponding HBLs and/or AWQC.

Kemira, On-Site Surface Impoundment Scenario

For the on-site surface impoundment scenario at Kemira, manganese was the only constituent detected in the SPLP at levels above the HBL; the exceedence is less than a factor of 1.3. Zinc and nickel were detected above the AWQC at factors of 2.5 and 2.7, respectively. This surface impoundment is less than 100 ft from the Savannah River. There are no groundwater receptors in the vicinity. Any groundwater intercepting a plume from this impoundment discharges into the river, resulting in a significant DAF which clearly would be many orders of magnitude greater than the lowest AWQC exceedence factor of 2.5. Therefore, it was determined that the surface impoundment scenario at Kemira does not pose a threat to human health or the environment and no further risk assessment was necessary.

Millennium, Landfill Scenario

For the landfill scenario (Millennium Baltimore), antimony and vanadium were the constituents of concern detected in the SPLP above the HBL. The constituents of concern detected above the AWQC were aluminum, copper, lead, manganese, and zinc. Because the landfill is located in an estuary, the lower of the freshwater or saltwater AWQC for the protection of aquatic life were used to screen against the toxicants in the sludge managed in the landfill. The landfill is located approximately 1,000 feet from the Patapsco River.

Because of residential areas in the vicinity of the landfill, a drinking water well scenario was assessed. See USGS map of facility and surrounding area in Appendix C. Groundwater flow is expected to be west to east toward the river. See *Update of the Hazardous Waste Groundwater Task Force; April, 1998*. Definitive flow direction studies, however, are not available. (The referenced study addresses an on-site surface impoundment, which is located about 1,000 feet from the landfill and immediately adjacent to the river). Millennium was not aware of any drinking water wells in the area. Although there no known groundwater receptors downgradient of the landfill, we modeled impacts on potential drinking water wells in the residential area to the southeast of the site (Swan Creek). Note that Susan Egan (MD Department of Public Works) confirmed that the Swan Creek community, located 2,500 ft south of the Millennium facility, is on a public water supply. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the full risk assessment.

The landfill scenario was also assessed for infiltration and dilution to the Patapsco River. The underlying soils for the on-site surface impoundment located near the landfill are characterized as clay and silt, except in the northeast quarter where the underlying sediment is a beach sand. However, we used the soils information from national data bases (e.g., STATSGO) to characterize the soils underlying the landfill. Using calculated infiltration rates to the river using the landfill model and the river flow rate, we calculated the DAF and compared it against the HBLs. The flushing rate for the river/estuary is reported to be 201 to 206 cubic meters per second according to Carl F. Cerco of the U.S. Army Corps of Engineers. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the full risk assessment.

Table 3.14 - Initial Screening Analysis for Sulfate Process Digestion Sludge

Constituent	MI-SO-02 (Landfill Scenario) SPLP (mg/L)	KP-SO-03 (Surface Impoundment Scenario) SPLP (mg/L)	HBL (mg/L)	AWQC (mg/L)
Aluminum	2.0	<0.01	16	0.087
Antimony	0.023	<0.005	0.0063	0.014
Barium	0.07	0.061	1.1	1
Boron	0.23	0.53	1.4	NA
Calcium	0.9	796	NA	NA
Chromium	0.17	<0.005	23	0.74
Cr +6	<0.02	<0.02	0.047	0.011
Cobalt	<0.005	<0.005	0.94	NA
Copper	0.37	<0.005	1.3	0.0031
Iron	12.0	<0.05	5	1
Lead	0.004 (1)	<0.005	0.015	0.0025
Magnesium	5.5	146	--	NA
Manganese	0.36	0.93	0.73	0.05
Mercury	<0.00011 (1)	<0.0002	0.0047	0.000050
Molybdenum	<0.005	<0.005	0.078	NA
Nickel	0.007	0.022	0.31	0.0082
Sodium	3.6	998	NA	NA
Thallium	<0.0022	<0.005	0.0013	0.0017
Tin	<0.01	<0.01	9	NA
Titanium	0.28	<0.005	NA	NA
Vanadium	0.42	<0.005	0.14	NA
Zinc	0.30	0.20	4.7	0.081

(1) Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.

NA- Not Available

3.2.6 Commingled Wastewaters from the Chloride and Sulfate Process

Waste Generation

As indicated above in Section 3.2.1, the two facilities that use both the chloride and sulfate processes to produce titanium dioxide, commingle the wastewaters generated at various points in the sulfate and the chloride production processes.

Chloride Process Wastewaters

At Kemira Pigments, the chloride process wastewaters that are included in this category are neutralized wastewaters from the scrubbing of the chlorinator off-gases and the product finishing wastewater. The Millennium Inorganic Chemicals (Baltimore, MD) chloride process wastewaters include the acidic digestion scrubber wastewaters and wastewater from purification of titanium tetrachloride generated during chlorination (coke and ore/acid mixture).

Sulfate Process Wastewaters

At Kemira, the sulfate process wastewaters include the wastewaters from scrubbing of gases produced during digestion, evaporator condensate from the precipitation unit, the calciner scrubber wastewater, the sulfate waste sludge settling pond supernatant (as described above in Section 3.2.5) and product finishing wastewaters. At Millennium Inorganic Chemicals (Baltimore, MD) sulfate process wastewaters include the calciner scrubber wastewater and finishing wastewaters.

Waste Management Practices

Both facilities commingle the wastewaters described above prior to being treated in their on-site wastewater treatment systems.

Kemira Pigments, Inc.

At Kemira, the wastewater treatment system is comprised of a concrete neutralization tank followed by a series of unlined settling ponds. The effluent from the settling ponds is discharged via a permitted NPDES outfall.

Millennium Inorganic Chemicals, Inc.

At Millennium Inorganic Chemicals (Baltimore, MD) the wastewater treatment system consists of tanks for neutralization and a series of unlined settling ponds. The effluent from the settling ponds is discharged via a permitted NPDES outfall.

Table 3.15 presents the wastewaters that are managed in the unlined units that are a part of the wastewater treatment systems at each facility.

Table 3.15 - Commingled Wastewaters from the Chloride and Sulfate Process		
Facility	Wastewater (RIN #)	Total Volume (MT/yr)
Kemira Pigments	Digestion Scrubber Purge (RIN 1)	298,000

Facility	Wastewater (RIN #)	Total Volume (MT/yr)
	Primary Pond Effluent/Sulfate Sludge Pond (RIN 3)	325,000
	Evaporator Condensate (RIN 4)	7,945,000
	Calciner Scrubber (RIN 5)	298,000
	Neutralized chloride acid (RIN 17) ²²	831,000
	Finishing wastewater (RIN 9)	3,150,000
Millennium Inorganic Chemicals; Baltimore, MD	Purification Wastewater (RIN 1)	1,578,120
	Scrubber Wastewater/HCl (RIN 2)	4,536
	Calciner Scrubber Wastewater (RIN 8)	1,380,781
	Finishing Wastewater (RIN 13)	373,594

Waste Characterization

Samples of this waste were collected at both the Millennium and Kemira facilities. At Kemira Pigments, Inc., Sample KP-WW-01 was collected at the point where the weir discharges the effluent into the first settling pond. At the Millennium facility in Baltimore, Sample MI-WW-04 was collected of the treated wastewater from the lime neutralization process of the wastewater treatment system upstream of the first settling pond.

Totals and SPLP filtrate analyses were conducted on the samples. The samples contained high levels of solids as a result of the facilities’ practice of mixing waste solids and wastewaters in the same unit. To isolate the impact of the wastewater on the environment from that of the sludge, we conducted the SPLP on the waste matrix, and separately analyzed the filtrate and the leachate generated from the leaching step.

A summary of the analytical data results are presented in Appendix A. Detailed reports of the record sampling trips, the complete set of analytical data and the validation reports is available in the “Sampling and Analytical Data Report For Record Sampling and Characterization of Wastes from the Inorganic Titanium Dioxide Manufacturing Sector” for Millennium Inorganics (Baltimore, MD) and Kemira Pigments. These reports are available in the docket for this rule.

Results of Screening Analysis

The management of these commingled wastewaters in the unlined units that make up the wastewater treatment systems were evaluated. **Table 3.16** presents the analytical results for the

²²Includes “Chloride Waste Acid” (RIN 7) volume.

constituents found to be present in either of the SPLP filtrates at levels exceeding the HBLs and/or AWQC.

At Kemira, the two unlined final impoundments of the wastewater treatment system were screened using the SPLP filtrate results from Sample KP-WW-01. The Kemira Pigments, Inc. impoundment screened out since no constituents were detected above the HBLs or AWQC. Therefore, no further risk assessment was required.

At the Millennium facility in Baltimore, the unlined settling pond that is part of the wastewater treatment plant was assessed for exposure using the SPLP filtrate results for Sample MI-WW-04. The filtrate is the closest approximation of the mobile portion of the wastewater likely to leach out of the bottom of the unlined surface impoundment. The constituents detected above the HBL were manganese and arsenic. The constituents detected above the AWQC were arsenic, manganese and nickel. The surface impoundment at the Millennium facility in Baltimore was assessed for potential drinking water well and surface water contamination. See *Section 3.2.5 Sulfate Process Digestion Sludge* for discussion regarding potential drinking water wells at this facility. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the full risk assessment.

Table 3.16 - Initial Screening Analysis for Commingled Wastewaters from the Chloride and Sulfate Process				
Constituent	KP-WW-01 SPLP filtrate (mg/L)	MI-WW-04 SPLP filtrate (mg/L)	HBL (mg/L)	AWQC (mg/L) freshwater/saltwater
Aluminum	<0.1	<0.1	16	0.087
Antimony	<0.005	<0.005	0.0063	0.014
Arsenic	<0.005	<0.005	0.0007 4	0.000018
Barium	0.11	0.49	1.1	NA
Beryllium	<0.002	<0.002	0.031	NA
Boron	0.86 B	0.40	1.4	NA
Cadmium	<0.005	<0.005	0.0078	0.0022
Calcium	95.2	1,430	NA	NA
Chromium	0.33	<0.005	23	0.74
Cobalt	<0.005	<0.005	0.94	NA
Copper	<0.005	<0.005	1.3	0.0090/0.0031
Iron	<0.05	<0.05	5	1
Lead	<0.003	<0.003	0.015	0.0025

Table 3.16 - Initial Screening Analysis for Commingled Wastewaters from the Chloride and Sulfate Process				
Constituent	KP-WW-01 SPLP filtrate (mg/L)	MI-WW-04 SPLP filtrate (mg/L)	HBL (mg/L)	AWQC (mg/L) freshwater/saltwater
Magnesium	180	152	NA	NA
Manganese	<0.005	9.95	0.73	0.05
Mercury	<0.0002	<0.0002	0.0047	0.000050
Molybdenum	0.045	0.006	0.078	NA
Nickel	<0.005	0.011	0.31	0.052/ 0.0082
Potassium	129	17.3	NA	NA
Silver	<0.001	<0.001	0.078	0.0034 /0.0019
Sodium	236	661	NA	NA
Thallium	<0.005	0.004	0.0013	0.0017
Tin	<0.01	<0.01	9.4	NA
Titanium	<0.005	<0.005	NA	NA
Vanadium	0.045	<0.005	0.14	NA
Zinc	<0.05	0.072	4.7	0.12 /0.081

NA- Not Available

*Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.

3.2.7 Wastewater Treatment Sludges from Commingled Chloride and Sulfate Process Wastewaters

Waste Generation and Management

This waste is generated at the two facilities that use both the chloride and sulfate processes to produce titanium dioxide. As indicated above, these facilities commingle the chloride process solids associated with the titanium tetrachloride portion of the titanium dioxide manufacturing process, which are Bevill-exempt mineral processing wastes under the Bevill exemption, with other non-Bevill-exempt wastewaters in the facility’s wastewater treatment system. After commingling of the Bevill-exempt solids with the non-Bevill-exempt wastewaters, the resulting solids from the commingled wastewaters contain a mixture of Bevill-exempt and non-Bevill-exempt wastewater treatment solids. Data provided by these two facilities show that this waste contains at least 35 percent non-Bevill-exempt solids. **Table 3.17** shows the calculation deriving

the percent solids from the various wastewater streams based on the reported information for the Millennium facility in Baltimore, MD. The percent solids for the Kemira facility are expected to contain a similarly high percentage of non-exempt solids (perhaps higher) because Kemira's exempt solids from the reactor are managed in a dedicated unit, and because Kemira (unlike Millennium) also commingles their sulfate process digestion scrubber wastes.

Table 3.17 - Estimation of Non-Exempt Solids Contribution to Wastewater Treatment Sludges from Commingled Chloride and Sulfate Process Wastewaters at Millennium Baltimore		
Waste (RIN #)	Volume (MT/yr)	Estimated Solids Loading (MT/yr)
<u>Wastewaters bearing Bevill-exempt Solids*</u>		
Unreacted coke and ore, waste acids (RIN 1)	1,578,120	154,656
Chloride process air emission scrubber (RIN 2)	4,536	667
<u>Wastewaters bearing non-Bevill-exempt solids**</u>		
Air emissions scrubber (sulfate process) (RIN 8)	380,781	69,039
Finishing wastewater (sulfate process) (RIN 13)	373,594	18,680
Totals	3,337,031	243,042
Percent non-Bevill-exempt solids = $[(69,039+18,680)/243,042] = 36\%$		

*Calculated using % solids measured during EPA's record sampling (see Analytical Data Report)

**Calculated using % solids reported in Table III.1 of RCRA 3007 Survey

At Kemira Pigments, Inc., all of the wastewaters generated during the production of titanium dioxide via the sulfate and chloride process are sent to the wastewater treatment system. A sludge is generated in the final settling pond of the facility's wastewater treatment system. The solids are dredged from this impoundment, filtered using a filter press, placed in piles for drainage and then sent to an on-site industrial landfill. At Millennium Inorganics (Baltimore, MD), the sludge is also dredged from the wastewater treatment system settling impoundment, filter pressed, and then placed in an on-site industrial landfill.

Table 3.18 presents the management of the wastewater treatment sludges from commingled chloride and sulfate process wastewaters at each facility.

Table 3.18 - Waste Management Practices and Volumes for Wastewater Treatment Sludges from Commingled Chloride and Sulfate Process Wastewaters		
Facility (RIN #)	Management Practice	Total Volume (MT/yr)
Millennium Inorganic Chemical Plant, Baltimore, Maryland (RIN 4)	Dredged from impoundment, filter pressed, placed in on-site industrial landfill	93,121
Kemira Pigments, Inc. (RIN 14)	Dredged from impoundment, filter pressed, drainage piles, placed in on-site industrial landfill	66,000

Waste Characterization

This waste was characterized using samples collected from the Kemira and Millennium Baltimore facilities. At Kemira, Sample KP-SO-02 was collected as a composite sample from the waste pile immediately after the filter press. At the Millennium facility in Baltimore, Sample MI-SO-01 was collected immediately after the filter press as a composite sample of eight scoops from the sludge pile, prior to the sludge being transferred to the on-site landfill.

Totals, TCLP, and SPLP analyses were conducted on both samples. **Table 3.19** presents the analytical results for the constituents detected in the filtrate and the corresponding HBLs and/or AWQC. A summary of the analytical results for each sample is presented in Appendix A. Detailed reports of the record sampling trips, the complete set of analytical data, and the validation reports are available in the “Sampling and Analytical Data Report For Record Sampling and Characterization of Wastes from the Inorganic Titanium Dioxide Manufacturing Sector” for Millennium Inorganics (Baltimore, MD) and Kemira Pigments. These reports are available in the docket for this rulemaking.

Results of Initial Screening Analysis

Summary

At Kemira the exposure pathways were found not to present a risk under the initial screening analysis and required no further assessment. The landfill at the Millennium facility in Baltimore required further assessment beyond the initial screening analysis for infiltration of landfill leachate to the river pathway and the groundwater contamination pathway due to exceedences of both the HBL and AWQC.

Kemira Facility

The SPLP results for Sample KP-SO-02 were used to screen the pile and landfill scenario at Kemira Pigments, Inc. for impacts to the groundwater and air pathways.

For the groundwater pathway from the piles and landfill, the SPLP results showed HBL exceedences for antimony, arsenic, molybdenum, and thallium. However, the only potential receptors are the Savannah and Wilmington Rivers and the adjacent marshlands. A review of

various land-use maps and groundwater flow directions, and interviews with local county and plant officials about drinking water sources for the nearest communities, has revealed there is no reason to believe that any potentially impacted drinking water wells exist.^{23,24} Even if such wells were to exist, none of the four potential constituents of concern require DAFs greater than three, and two of the four constituents of concerns are only being assessed at ½ the detection limit. The closest potential downgradient communities are 3,000 feet away from the management units. With respect to potential impacts on the rivers and adjacent marshes, again, none of the constituents of concern require DAFs greater than three to be below the AWQC. Considering these factors, it is assumed that the groundwater pathway does not present a risk under the initial screening analysis.

Assessment of the air pathway for the piles and landfill indicates that all constituent concentrations in Kemira's waste sample are below the SSLs except thallium which is only slightly greater than the soil ingestion HBL (a factor of 1.3 higher). Thus, the air pathway was not assessed further.

Millennium's Baltimore, Maryland Facility

The SPLP results for Sample MI-SO-01 were used to screen the landfill scenario at Millennium Baltimore for impacts to surface water and drinking water wells. The constituents detected in the SPLP above the AWQC were aluminum, arsenic, manganese, and thallium; manganese also exceeded the HBL. Therefore, further risk modeling assessment was required for the wastewater treatment sludges in the landfill scenario. Based on the distance of the landfill from the Patapsco River and the fact that the potential existence of drinking water wells to the southeast, the groundwater pathway cannot be ruled out and both scenarios required a full risk assessment. See *Section 3.2.5 Sulfate Process Digestion Sludge* for a discussion of the assumptions for this assessment. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the full risk assessment.

Assessment of the air pathway for the landfill indicates no significant risks are likely from particulate releases for several reasons. First, all constituents were below soil ingestion levels, except for manganese and vanadium, which exceed the soil ingestion levels by a factors of about 3. In both cases these constituents were below the air characteristic levels for waste piles shown in Table 3.19. The air characteristic levels calculated for landfills were an order of magnitude higher (20,000 mg/kg in both cases)²⁵ It is also highly unlikely that wind blown particulates from landfills would be significant due to the common usage of longer-term cover at landfills. Furthermore, the waste is generated and disposed of as a wet sludge, making the formation particulates less likely.

²³Phone log. Ron Josephson (EPA) to Jim McKirgan, Chatham County, GA Department of Public Works. December 22, 1999.

²⁴Maps are provided in Appendix C.

²⁵ U.S. Environmental Protection Agency, "Revised Risk Assessment for the Air Characteristic Study", EPA 530-R-99-019a, November 1999, Table 4.1 (Landfills)

Table 3.19 - Initial Screening Analysis for Wastewater Treatment Sludges from Commingled Chloride and Sulfate Process Wastewaters

Constituent	MI-SO-01 Totals (mg/kg)	MI-SO-01 SPLP (mg/L)	KP-SO-02 Totals (mg/kg)	KP-SO-02 SPLP (mg/L)	HBL (mg/L)	AWQC (mg/L)	SSL(1) (mg/kg)
Aluminum	8,740	0.24	4,520	<0.1	16	0.087	47,000
Antimony	0.6	0.006	0.8	0.013	0.0063	0.014	32*
Arsenic	1.6	0.00005 (2)	2.4	<0.0035	0.00074	0.000018	5
Barium	49.8	0.06	40.2	0.078	1.1	1	440
Beryllium	1.5	<0.002	0.4	<0.002	0.031	NA	160*
Boron	9.4	0.15	16.9	0.28	1.4	NA	26
Chromium	1,230	0.001 (2)	712	0.001 (2)	23	0.74	120,000*
Cobalt	2.6	<0.005	2.4	<0.005	0.94	NA	6.7
Copper	16.7	<0.002	12.6	0.004	1.3	0.0031	17
Iron	62,700	<0.05	36,500	<0.05	5	1	430,000*
Lead	1.3	0.002 (2)	0.002	0.001 (2)	0.015	0.0025	400*
Manganese	12,700	2.63	3,130	<0.005	0.73	0.05	3,800*(3)
Molybdenum	1.6	0.013	10.6	0.093	0.078	NA	400*(4)
Nickel	59.9	<0.005	47.1	<0.005	0.31	0.0082	1,600*
Silver	0.6	<0.001	1.5	<0.001	0.078	0.0019 sw AWQC	400*
Thallium	3.0	0.003 (2)	8.1	<0.0022	0.0013	0.0017	6.4 (4)
Tin	2.7	<0.01	75.2	<0.01	9	NA	48,000*
Titanium	5,270	<0.005	8,310	<0.005	NA	NA	NA
Vanadium	2,320	0.004 (2)	1,570	0.039	0.14	NA	720 (5)
Zinc	2.9	0.025 (2)	31.5	0.02 (2)	4.7	0.08	24,000*
Dioxins/Furans (ng/kg for totals and ng/L for SPLP)							

Table 3.19 - Initial Screening Analysis for Wastewater Treatment Sludges from Commingled Chloride and Sulfate Process Wastewaters

Constituent	MI-SO-01 Totals (mg/kg)	MI-SO-01 SPLP (mg/L)	KP-SO-02 Totals (mg/kg)	KP-SO-02 SPLP (mg/L)	HBL (mg/L)	AWQC (mg/L)	SSL(1) (mg/kg)
2378-TCDF (TEF=0.1) ²⁶	1710	<0.010	2.2	<0.010	NA	NA	NA
Total TCDF	2,530	<0.010	21.5	<0.010	NA	NA	NA
2378-TCDD (1.0)	<0.3	<0.010	<0.3	<0.010	NA	NA	NA
Total TCDD	1.9	<0.010	<0.3	<0.010	NA	NA	NA
12378-PeCDF (0.05)	6,580	<0.051	3.3	<0.052	NA	NA	NA
23478 PeCDF (0.5)	921	<0.051	2.0	<0.052	NA	NA	NA
Total PeCDF	9,870	<0.051	8.5	<0.052	NA	NA	NA
12378-PeCDD (1.0)	<1.6	<0.051	<1.3	<0.052	NA	NA	NA
Total PeCDD	<1.6	<0.051	<1.3	<0.052	NA	NA	NA
123478 HxCDF (0.1)	12,200	<0.051	4.7	<0.052	NA	NA	NA
123678 HxCDF (0.1)	1,890	<0.051	<1.3	<0.052	NA	NA	NA
234678 HxCDF (0.1)	102	<0.051	<1.3	<0.052	NA	NA	NA
123789 HxCDF (0.1)	1,380	<0.051	1.7	<0.052	NA	NA	NA
Total HxCDF	18,100	<0.051	6.3	<0.052	NA	NA	NA
123478-HxCDD (0.1)	<16.1	<0.051	<1.3	<0.052	NA	NA	NA
123678-HxCDD (0.1)	<16.1	<0.051	<1.3	<0.052	NA	NA	NA
123789-HxCDD (0.1)	<16.1	<0.051	<1.3	<0.052	NA	NA	NA

²⁶TEF=Toxicity Equivalent Factor, provided in parentheses following congener name. Dioxin TEQs calculated using WHO-TEFs.

Table 3.19 - Initial Screening Analysis for Wastewater Treatment Sludges from Commingled Chloride and Sulfate Process Wastewaters							
Constituent	MI-SO-01 Totals (mg/kg)	MI-SO-01 SPLP (mg/L)	KP-SO-02 Totals (mg/kg)	KP-SO-02 SPLP (mg/L)	HBL (mg/L)	AWQC (mg/L)	SSL(1) (mg/kg)
Total HxCDD	<16.1	<0.051	<1.3	<0.052	NA	NA	NA
1234678 HpCDF (0.01)	3,620	<0.051	2.0	<0.052	NA	NA	NA
1234789 HpCDF (0.01)	5,920	<0.051	3.0	<0.052	NA	NA	NA
Total HpCDF	11,500	<0.051	6.6	<0.052	NA	NA	NA
1234678-HpCDD (0.01)	<16.1	<0.051	<1.3	<0.052	NA	NA	NA
Total HpCDD	<16.1	<0.051	<1.3	<0.052	NA	NA	NA
OCDF (0.0001)	22,000	<0.100	55	<0.100	NA	NA	NA
OCDD (0.0001)	<197	0.110	3.3	<0.100	NA	NA	NA
2378-TCDD TEQ	2,615	0.00011 ng/L or 0.11 ppq	2.08	ND	0.0071 ng/L	0.0031 ng/L	45 ng/kg* (6)

NA- Not Available

(1) Soil screening levels (SSLs) are based on soil background, except where soil ingestion levels are otherwise noted by the symbol “*”; in all cases the background levels are equivalent to or below soil ingestion levels. Air characteristic levels are noted when soil ingestion levels are exceeded. See “*Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes* (August 2000) in the docket for details and sources of the HBLs and SSLs.

(2) Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.

(3) The air characteristic level is 3,000 mg/kg at 25m and drops to 30,000 at 150m.

(4) An air characteristic level has not been determined.

(5) The air characteristic level is 4,000 mg/kg at 25 m.

3.2.8 Waste Acid (Ferric Chloride) from the Chloride-Ilmenite Process

Waste Generation and Management

This waste is generated at all three facilities that use the chloride-ilmenite process in the production of titanium dioxide. The ferric chloride waste acid is generated after solids are removed from the acidified solution of metal-chloride solids, unreacted coke and ore solids, and impurities formed during the initial chlorination reaction.

The DuPont facilities in Mississippi and Tennessee generate the majority of their Bevill-exempt solids from the filtration of this waste acid. The DuPont facility in Mississippi disposes of its ferric chloride waste acid in an on-site underground injection well. The DuPont facility in Tennessee recycles a portion of this waste back to the reaction and uses the remaining portion in the production of sodium chloride (NaCl). At this facility, an iron carbonate (FeCO₃) waste is generated as a result of the NaCl production. As discussed in Section 3.3, this FeCO₃ residual was not evaluated further.

The DuPont facility in Delaware has a slightly different process. The majority of their Bevill-exempt solids are generated prior to the generation of the acid. Once the waste acid is removed from the product stream, this facility adds a processing chemical to their waste acid, removes solids, and stores the acid in tanks (as well as in an impoundment when their tank capacity is exceeded). The waste acid is then marketed for use as a wastewater and drinking water treatment reagent. **Table 3.20** presents the management of this waste at each facility.

Table 3.20 - Waste Management Practices and Volumes for Waste Acid (ferric chloride) from the Chloride-Ilmenite Process		
Facility	Management	Total Volume
E.I. DuPont de Nemours; DeLisle Plant; Pass Christian, MS (RIN 5)	On-site hazardous waste underground injection well	1,035,869 MT/yr
E.I. DuPont de Nemours; New Johnsonville, TN	Used on-site in the production of NaCl	791,840 MT/yr
E.I. DuPont de Nemours; Edge Moor, DE	Storage in tanks and surface impoundment prior to sales	60,000-70,000 dry ton at <40% concentration (~148,000 MT/yr)

Waste Characterization

The DuPont facility in Mississippi reported D002, D007 and D008 waste codes for this waste in their RCRA §3007 questionnaire. All three generators also reported this waste with pH levels of 1 or less. One sample collected during record sampling was used to characterize this waste. Sample DPE-WW-03 was collected at the influent to the storage pond at the DuPont Edge Moor facility during record sampling.

EPA conducted totals and SPLP analyses on Sample DPE-WW-03. There was not sufficient sample volume to conduct TCLP analysis of EPA’s sample. A summary of EPA’s analytical results is presented in **Table 3.21** and **Appendix A**. Detailed reports of the record sampling trips, the complete set of analytical data, and the validation reports are available in the “Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes From the Inorganic Titanium Dioxide Manufacturing Sector” for the DuPont Edge Moor facility. This report is available in the docket for this rulemaking.

Results of Screening Analysis

EPA determined that this waste does not warrant listing because of the characteristic nature of this waste and the adequacy of existing enforcement authorities under Subtitle C to ensure proper management of this waste. Note, however, that, as described above, EPA did collect a sample of this material during our field investigation (see **Table 3.21**).

As detailed below, all three generators of the ferric chloride waste acid acknowledge the hazardous nature of this waste.

DuPont DeLisle Plant; Pass Christian, MS

The DuPont facility in Mississippi disposes of this waste via deep well injection onsite and assigned three separate characteristic codes to this material (D002, D007, D008). In their survey response, the Mississippi facility reported TCLP results for chromium as 443 mg/L and for lead as 167 mg/L²⁷. The Mississippi facility manages its ferric chloride via a permitted Class I injection well. DuPont's no-migration petition for the injection wells was approved by EPA Region IV via a letter dated May 5, 2000. There is no land placement of the material prior to injection. The Safe Drinking Water Act provides regulatory control of the deep well injection scenario.

DuPont New Johnsonville, TN Facility

The Tennessee facility uses the waste acid (RIN 10) to manufacture sodium chloride (NaCl), generating a FeCO₃ waste stream. DuPont characterized RIN 10 as having a pH of 1 and total chromium levels of 144 mg/kg and total lead levels of 64.9 mg/kg.

As discussed in Section 3.3.2, the FeCO₃ waste stream did not warrant further assessment because it is generated from an out-of scope production process. The use of ferric chloride in the production of NaCl was not investigated in depth because there was no known exposure route associated with the management of the material prior to inserting it into a non-consent decree production process. Note, however, that EPA did collect a sample of the ferric carbonate residual (DPN-SO-03) during our field investigation. The results of the ferric carbonate analyses are provided in **Table 3.22**.

DuPont Edge Moor, DE Facility

Sample DPE-WW-03 was collected at the Delaware facility prior to placement of this material on the land. Our pH analysis showed a pH of less than 1.0. The facility's split sample results showed TCLP results of 415 mg/L for chromium and 49 mg/L for lead.

²⁷The TC standards for both chromium and lead is 5 mg/L.

Table 3.21 - Summary of Analytical Results for Waste Acid (ferric chloride) from the Chloride-Ilmenite Process

Constituent	DPE-WW-03 (mg/L)			DuPont Split Sample		HBL (mg/L)	AWQC (mg/L)
	Totals	SPLP Filtrate	SPLP Leachate	Totals (mg/L)	TCLP (mg/L)		
Aluminum	2,090	2,360	77.2	2,520		16	0.087
Antimony	2.26	1.8	<0.5	<50		0.0063	0.014
Arsenic	<2.5	<3.5	<0.5	<0.25	<20	0.00074	0.000018
Barium	15.5	19.6	15.6	13.9	16.8	1.1	1
Beryllium	<2	<0.24	<0.2	<2.5		0.031	NA
Boron	2.32	10.3	<5	<13		1.4	NA
Cadmium	5.09	3.03	<0.5	<10	<2.0	0.0078	0.0022
Calcium	94.3	310	16.8	53.8		NA	NA
Chromium	101	113	3.76	119	415	23	0.74
Chromium, +6	<0.2	NR			43	0.047	0.011
Cobalt	8.44	6.86	<0.5	7.44		0.94	NA
Copper	29.2	27.1	1.56	15.9		1.3	0.0090
Iron	164,000	175,000	5,310	[cbi]		5	1
Lead	62.2	76.4	2.66	70.9	49	0.015	0.0025
Magnesium	245	385	12.2	150		NA	NA
Manganese	1,770	1,790	51.6	1,870		0.73	0.05
Mercury	0.021	0.0007	0.0002	<0.020	<0.020	0.0047	0.000050

Table 3.21 - Summary of Analytical Results for Waste Acid (ferric chloride) from the Chloride-Ilmenite Process

Constituent	DPE-WW-03 (mg/L)			DuPont Split Sample		HBL (mg/L)	AWQC (mg/L)
	Totals	SPLP Filtrate	SPLP Leachate	Totals (mg/L)	TCLP (mg/L)		
Molybdenum	31	34.6	1.48	16.8		0.078	NA
Nickel	6.0	10.8	<0.5	5.9		0.31	0.052
Selenium	<25	4.43	<0.5			0.078	0.0050
Silver	1.14	2.58	0.93	5.16	<4.0	0.078	0.0034
Thallium	2.7	7.30	<0.5	<20		0.0013	0.0017
Tin	<25	6.2	<1	6.53		9.4	NA
Titanium	6,220	7,570	495	[cbi]		NA	NA
Vanadium	1,070	1,140	31.6	1,170		0.14	NA
Zinc	98.3	145	15.6	129		4.7	0.12
Dioxins/Furans (ng/L)*							
2,3,7,8-TCDF (TEF=0.1)	0.13	<0.11 ng/L	<0.12			NA	NA
Total TCDF	0.13	<0.11	<0.12			NA	NA
2378-TCDD (TEF=1)	<0.10	<0.11	<0.12			NA	NA
Total TCDD	<0.10	<0.11	<0.58			NA	NA
12378-PeCDF (0.05)	0.65	<0.55	<0.58			NA	NA
23478-PeCDF (0.5)	<0.50	<0.55	<0.58			NA	NA
Total PeCDF	0.65	<0.55	<0.58			NA	NA

Table 3.21 - Summary of Analytical Results for Waste Acid (ferric chloride) from the Chloride-Ilmenite Process

Constituent	DPE-WW-03 (mg/L)			DuPont Split Sample		HBL (mg/L)	AWQC (mg/L)
	Totals	SPLP Filtrate	SPLP Leachate	Totals (mg/L)	TCLP (mg/L)		
12378-PeCFD (TEF=1)	<0.50	<0.55	<0.58			NA	NA
Total PeCDD	<0.50	<0.55	<0.58			NA	NA
123478-HxCDF (0.1)	7.70	<0.55	<0.58			NA	NA
123678-HxCDF (0.1)	<0.50	<0.55	<0.58			NA	NA
234678-HxCDF (0.1)	<0.50	<0.55	<0.58			NA	NA
123789-HxCDF (0.1)	<0.50	<0.55	<0.58			NA	NA
Total HxCDF	8.2	<0.55	<0.58			NA	NA
123478-HxCDD (0.1)	<0.50	<0.55	<0.58			NA	NA
123678-HxCDD (0.1)	<0.50	<0.55	<0.58			NA	NA
123789-HxCDD (0.1)	<0.50	<0.55	<0.58			NA	NA
Total HxCDD	<0.50	<0.55	<0.58			NA	NA
1234678-HpCDF (0.01)	5.3	<0.55	<0.58			NA	NA
1234789-HpCDF (0.01)	5.00	<0.55	<0.58			NA	NA
Total HpCDF	13.0	<0.55	<0.58			NA	NA
1234678-HpCDD (0.01)	<0.50	<0.55	<0.58			NA	NA
Total HpCDD	<0.50	<0.55	<0.58			NA	NA
OCDF (0.0001)	1,200	1.3	<1.2			NA	NA

Table 3.21 - Summary of Analytical Results for Waste Acid (ferric chloride) from the Chloride-Ilmenite Process

Constituent	DPE-WW-03 (mg/L)			DuPont Split Sample		HBL (mg/L)	AWQC (mg/L)
	Totals	SPLP Filtrate	SPLP Leachate	Totals (mg/L)	TCLP (mg/L)		
OCDD (0.0001)	<1.0	<1.1	<1.2			NA	NA
2378 TCDD TEQ **	1.038 ng/L	0.00013 ng/L	ND			0.0071 ng/L	0.0031 ng/L

NA- Not Available

* TEF = Toxicity Equivalent Factor

** Toxicity Equivalents (TEQ) calculated using WHO-TEFs.

Table 3.22 - Summary of Analytical Results for Ferric Carbonate

Constituent	DPN-SO-03			HBL (mg/L)	AWQC (mg/L)
	Total (mg/kg)	TCLP (mg/L)	SPLP (mg/L)		
Aluminum	15,600	<1	0.17	16	0.087
Antimony	<0.5	<0.5	0.009	0.0063	0.014
Arsenic	2.1	<0.5	<0.0035	0.00074	0.000018
Barium	97.5	1.7	0.30	1.1	1
Beryllium	1.4	<0.02	<0.002	0.031	NA
Boron	<10	<2	0.21	1.4	NA
Cadmium	2.0	<0.05	<0.005	0.0078	0.0022
Calcium	12,300	59.9	2.2		
Chromium	3,040	0.01	<0.005	23	0.74
Chromium, +6	NA	<0.02	0.047	0.047	0.011
Cobalt	6.1	<0.05	<0.005	0.94	NA
Copper	41.2	<0.25	0.007	1.3	0.0090
Iron	180	<0.05	5	5	1
Lead	46.0	0.02	0.002	0.015	0.0025
Magnesium	13,100	120	24.1		
Manganese	30,200	59.4	0.18	0.73	0.05
Mercury	<0.1	<0.002	<0.0002	0.0047	0.000050
Molybdenum	7.2	<0.2	0.12		
Nickel	138	1.1	0.002	0.31	0.052
Selenium	1.5	<0.5	<0.005	0.078	0.0050
Silver	3.9	<0.1	<0.001	0.078	0.0034
Thallium	<2	<2	<0.005	0.0013	0.0017
Tin	<1	<0.5	<0.01		
Titanium	10,000	0.04	0.002		
Vanadium	6,690	0.05	0.14	0.14	NA
Zinc	<5	0.54	0.041	4.7	0.12

Table 3.22 - Summary of Analytical Results for Ferric Carbonate

Constituent	DPN-SO-03			HBL (mg/L)	AWQC (mg/L)
	Total (mg/kg)	TCLP (mg/L)	SPLP (mg/L)		
Dioxins/Furans (ng/L)					
2,3,7,8-TCDF (TEF=0.1)	<0.7	n/a	<0.01		
Total TCDF	44.9	n/a	<0.01		
2378-TCDD (TEF=1)	<0.7	n/a	<0.01		
Total TCDD	<0.7	n/a	<0.01		
12378-PeCDF (0.05)	46.2	n/a	<0.052		
23478-PeCDF (0.5)	11.7	n/a	<0.052		
Total PeCDF	193	n/a	<0.052		
12378-PeCDD (TEF=1)	<3.4	n/a	<0.052		
Total PeCDD	<3.4	n/a	<0.052		
123478-HxCDF (0.1)	235	n/a	<0.052		
123678-HxCDF (0.1)	26.2	n/a	<0.052		
234678-HxCDF (0.1)	10.4	n/a	<0.052		
123789-HxCDF (0.1)	<3.4	n/a	<0.052		
Total HxCDF	518	n/a	<0.052		
123478-HxCDD (0.1)	<3.4	n/a	<0.052		
123678-HxCDD (0.1)	<3.4	n/a	<0.052		
123789-HxCDD (0.1)	<3.4	n/a	<0.052		
Total HxCDD	<3.4	n/a	<0.052		
1234678-HpCDF (0.01)	<3.4	n/a	<0.052		
1234789-HpCDF (0.01)	<3.4	n/a	<0.052		
Total HpCDF	<3.4	n/a	<0.052		
1234678-HpCDD (0.01)	<3.4	n/a	<0.052		
Total HpCDD	<3.4	n/a	<0.052		
OCDF (0.0001)	7,590	n/a	<0.1		
OCDD (0.0001)	10.4	n/a	<0.1		

Table 3.22 - Summary of Analytical Results for Ferric Carbonate

Constituent	DPN-SO-03			HBL (mg/L)	AWQC (mg/L)
	Total (mg/kg)	TCLP (mg/L)	SPLP (mg/L)		
2378 TCDD TEQ **	39.3	n/a	ND	0.0071 ng/L	0.0031 ng/L

n/a = TCLP analysis for Dioxins/Furans not performed.

3.2.9 Non-Bevill-exempt Nonwastewaters from the Chloride-Ilmenite Process

Waste Generation and Management

Non-Bevill-exempt nonwastewaters are generated in several different ways at the three chloride-ilmenite facilities. All three DuPont facilities that produce titanium dioxide via the chloride-ilmenite process commingle their process wastewaters and subsequently generate wastewater treatment solids that fall within the non-Bevill-exempt nonwastewater category. This waste category consists of solids that drop out in on-site settling ponds or are filtered out of the treated wastewater prior to discharge. As discussed below, the wastewater treatment solids contain both Bevill-exempt and non-Bevill-exempt components.

Non-Bevill-exempt nonwastewaters may be generated at several other places in the chloride-ilmenite process. In addition, solids removed from ferric chloride waste acid at the Edge Moor site after the end of mineral processing are also not Bevill exempt and are covered by this waste category.

DuPont Edge Moor, DE Facility

The facility commingles several sources of solids and markets the mixture as Iron Rich®, also described as Solids Co-Product I, or SCP I. DuPont describes this material as “soil for landfill capping” and reports annual generation of 120,000 - 140,000 short tons. DuPont has asserted a variety of end uses for the Iron Rich®.²⁸ The predominant recent use has been for the construction of dikes to contain dredged river sediments at various Corp of Engineer disposal facilities in the vicinity of the plant. This scenario was assessed as comparable to an industrial Subtitle D landfill scenario. The Iron Rich® has also been used as daily cover at a municipal landfill (demonstration project) and as final cover for a closed onsite landfill. Other proposed uses include use as subsidence fill at a closed municipal landfill, structural fill by the local Port Authority, surcharge for road bed compaction, and construction of a wildlife refuge at the site of a closed landfill.

²⁸See Letter from Jonathan R. Bacher, Manager, Ash marketing, VFL Technology Corporation, to Ms. Nancy Marker, Solid Waste Management Branch - DNREC, re DNREC Approval SWA-95/29, DuPont Iron-Rich Filter Cake, dated July 27, 1999

The primary components of this waste are:

1. “Iron-containing metal chlorides and coke/ore solids” from the Reaction Area (80%) that result from the neutralization of RIN 14 (“solids to neutralizer”) and RIN 4 (“metal chlorides with coke and ore solids”)
2. “Iron-containing metal chlorides and coke/ore solids from ... Solids Co-Product II area” (<10%) that results from the removal of solids from ferric chloride (i.e., SCP II)
3. “Iron hydroxide slurry from the HCl neutralizer and from the wastewater treatment system” (>10%), that results from wastewater treatment.

Each of these components of Iron Rich® contain differing proportions of Bevill-exempt and non-Bevill-exempt materials. The first category consists of the bulk of the Bevill-exempt solids generated by Edge Moor. The second category consists of solids removed from this facility’s ferric chloride waste acid stream. These solids are not Bevill-exempt because they are removed from the waste acid after mineral processing has ended and the production of ferric chloride has begun (marked by the addition of a processing chemical prior to solids removal). The third category, wastewater treatment solids, contains both Bevill-exempt and non-Bevill-exempt solids. **Table 3.23** summarizes the contribution of Bevill-exempt and non-Bevill-exempt solids to Edge Moor’s wastewater treatment solids.

Table 3.23 - Estimation of Non-Bevill Exempt Solids Contribution to DuPont Edge Moor’s Wastewater Treatment Solids		
Waste (RIN #)	Volume (MT/yr)	Estimated Solids Loading* (MT/yr)
<u>Wastewaters bearing Bevill-exempt Solids</u>		
Pre-treated reaction area scrubber (RIN 1)	10,521**	52.6
Coke and ore washwater (RIN 2)	97,611	9,761
Fume disposal/HCl scrubber (RIN 3)	169,609	3,392
Calcium chloride filtrate (RIN 8)	605,513	182
Purification scrubber (RIN 12)	1,769,305	0
<u>Wastewaters bearing non-Bevill-exempt solids</u>		
Chlorine scrubber water (RIN 7)	4,354	0
OVS scrubber water (RIN 9)	59,589	595
Oxidation and finishing wastewater (RINs 6 & 11)	4.53	<0.5***
Dryer scrubber wastewater (RIN 13)	124,259	621

Table 3.23 - Estimation of Non-Bevill Exempt Solids Contribution to DuPont Edge Moor's Wastewater Treatment Solids		
Waste (RIN #)	Volume (MT/yr)	Estimated Solids Loading* (MT/yr)
Totals	2,840,765	14,604
Percent non-Bevill-exempt solids = [(0+595+0.5+621)/14,604] = 8.3%		

*Calculated using % solids reported in Table III.1 of RCRA §3007 Survey

**RIN 1 volume of 70,110 MT/yr includes commingled RIN 9 volume of 59,589 MT/yr. For this analysis, the volumes are reported separately to reflect the potential for RIN 9 to contain non-Bevill-exempt solids subject to the listing.

***Loading estimate provided in August, 2000 correspondence from Angie Strzelecki, DuPont.

Overall, we estimate that the Iron Rich® is currently comprised of over 10% non-Bevill-exempt solids (<10% ferric chloride solids + 0.08* 10% WWT solids). This estimate is based on the facility's engineering estimates of solids content in their various untreated wastewaters (EPA does not have independent analytical results).

New Johnsonville, TN Facility

At the DuPont facility in Tennessee, non-Bevill-exempt nonwastewaters are predominantly associated with wastewater treatment sludge. **Table 3.24** summarizes the contributions of Bevill-exempt and non-Bevill-exempt solids to the Tennessee facility wastewater treatment solids.

Table 3.24 - Estimate of Non-Bevill Exempt Solids Contribution to DuPont New Johnsonville's Wastewater Treatment Solids		
Waste (RIN #)	Volume (MT/yr)	Estimated Solids Loading* (MT/yr)
<u>Wastewaters bearing Bevill-exempt Solids</u>		
Purification area scrubber water (RIN 3)	82,000	1
Maintenance scrubber (Reaction Area) (RIN 103)	11,112	<1
Recovered ore wastewater (RIN 4)	442,800	8,856
Pre-treated Reaction Area scrubber water (RIN 2)	246,000	36,900
Reaction startup scrubber (RIN 102)	3,268	<1
Reaction maintenance wastewater (RIN 109)	1,900	20
<u>Wastewaters bearing non-Bevill-exempt solids</u>		

Table 3.24 - Estimate of Non-Bevill Exempt Solids Contribution to DuPont New Johnsonville's Wastewater Treatment Solids		
Waste (RIN #)	Volume (MT/yr)	Estimated Solids Loading* (MT/yr)
Chlorine unloading scrubber water (RIN 101)	82	12
Maintenance washhouse scrubber water (RIN 105)	4,540	681
HCl Storage tank scrubber water (RIN 5)	9,110	<1
Hillside Pond wastewater** (RIN 106)	102,200	1,042
Finishing wastewaters (RINs 6, 111, & 113)	6,566,998	600 - 2,000***
Washhouse wash water (RIN 114)	454	5
Oxidation scrubber water (RIN 104)	6	1
Totals	7,470,470	48,818 +/- 700
Percent non-Bevill-exempt solids = $[(12+681+0+1042+1300+5+1)/48,818] = 6.2\%$ (+/- 1.3)		

*Calculated using % solids reported in Table III.1 of RCRA §3007 Survey.

**Contains Bevill-exempt and non-Bevill-exempt solids; breakdown not provided.

***Loading estimate provided in August, 2000 correspondence from Angie Strzelecki, DuPont.

This facility's wastewater treatment system includes a series of three unlined surface impoundments. The solids from the first two surface impoundments are dredged and placed in the "Hillside Pond" for de-watering. When this "Hillside Pond" is full, the solids are removed and placed in the facility's on-site landfill. These solids contain a mixture of Bevill-exempt and non-Bevill-exempt solids.

The Bevill-exempt coke and ore solids from filtering the iron chloride waste acid are placed in the same landfill as wastewater treatment sludge.

DuPont DeLisle Plant; Pass Christian, MS Facility

The DuPont facility in Mississippi generates wastewater treatment solids which contain non-Bevill-exempt solids. **Table 3.25** summarizes the contributions of Bevill-exempt and non-Bevill-exempt solids to the Mississippi facility's wastewater treatment solids.

Table 3.25 - Estimation of Non-Bevill Exempt Solids Contribution to DuPont DeLisle's Wastewater Treatment Solids		
Waste (RIN #)	Volume (MT/yr)	Estimated Solids Loading* (MT/yr)
<u>Wastewaters bearing Bevill-exempt Solids</u>		
Pre-treated reaction scrubber water (RIN 3)	328,802	49,320
Return water from Borrow Pit (RIN 109)	191,000	2
Wastewater from Reaction Emergency Scrubber (RIN 102)	15,536	2,331
<u>Wastewaters bearing non-Bevill-exempt solids</u>		
Chlorine unloading scrubber water (RIN 101)	202	30
HCl storage scrubber (RIN 9)	3,835	<1
Oxidation scrubber (RIN 6)	9,259	1,385
Oxidation and Finishing wastewater (RIN 8, 103, & 104)	2,859,842	200 - 600**
Totals	3,408,476	53,468 (+/- 200)
Percent non-Bevill-exempt solids = [(30+0+1385+400)/53,468] = 3.4% (+/- 0.4)		

*Calculated using % solids reported in Table III.1 of RCRA §3007 Survey

**Loading estimate provided in August, 2000 correspondence from Angie Strzelecki, DuPont.

The commingled wastewaters are managed in lined on-site surface impoundments. The wastewater treatment solids are dredged from the surface impoundments and placed in the "Borrow Pit area" at the facility. The solids are dredged periodically and placed in an on-site landfill. The Bevill-exempt coke and ore solids from filtering the iron chloride waste acid are placed in the same landfill as the wastewater treatment sludge.

Table 3.26 summarizes the management for this waste category at each facility. This table reflects current management of commingled non-Bevill-exempt and Bevill-exempt streams and provides a rough estimate of the portion of the commingled waste that is covered by the non-Bevill-exempt nonwastewater category.

Table 3.26 - Waste Management Practices and Volumes for Non-Bevill-exempt Nonwastewaters from the Chloride-Ilmenite Process			
Facility (RIN #)	Management Practices	Total Volume Combined Bevill exempt and non-Bevill exempt (MT/yr)	Estimated non-Bevill-exempt Volume (MT/yr)
E.I. DuPont de Nemours Edge Moor, DE (RIN 23)	Combines with other solids, stores in piles, and markets as “Iron Rich®”	117,936	12,800
E.I. DuPont de Nemours New Johnsonville, TN (RIN 108)	Wastewater treatment sludges de-watered and place in on-site landfill*	18,890	1,200
E.I. DuPont de Nemours DeLisle Plant; Pass Christian, MS (RIN 108)	Wastewater treatment sludges dredged from on-site surface impoundment, de-watered, and placed in on-site landfill*	17,237	600

*Volumes from solids removed from ferric chloride were not reported.

Waste Characterization

Two samples were collected during record sampling to characterize this waste. At the Delaware facility, Sample DPE-SO-01 was collected from the Iron Rich® dewatering operation just prior to truck loading operation for off-site transport to the customer. At the Tennessee facility, Sample DPN-SO-01 was collected directly from the “Hillside Pond.”

Totals, TCLP, and SPLP analyses were conducted on both samples. A summary of the analytical results for each sample is presented in Appendix A. Detailed reports of the record sampling trips, the complete set of analytical data, and the validation reports are available in the “Sampling and Analytical Data Report For Record Sampling and Characterization of Wastes from the Inorganic Titanium Dioxide Manufacturing Sector” for the DuPont Edge Moor, DE and New Johnsonville, TN facilities. These reports are available in the docket for today’s rulemaking.

Results of Screening Analysis

The SPLP results for Sample DPN-SO-01, and the total, SPLP, and TCLP results for Sample DPE-SO-01 were used for the initial screening analysis for this waste. **Table 3.27** presents the constituents detected in the SPLP analysis of Sample DPN-SO-1 and the TCLP analysis for Sample DPE-SO-01 and the associated HBLs and AWQC for the constituents.

Table 3.27 - Initial Screening Analysis for Non-Bevill-exempt Nonwastewaters from the Chloride-Ilmenite Process

Constituent	DPE-SO-01 Total (mg/kg)	DPE-SO-01 TCLP (mg/L)	DPE-SO-01 SPLP (mg/L)	DPN-SO-01 Total (mg/kg)	DPN-SO-01 SPLP (mg/L)	HBL (mg/L)	AWQC (mg/L)	SSL (1) (mg/kg)
Aluminum	10,100	<1	<0.1	5,770	<0.1	16	0.087	47,000
Antimony	0.9	<0.021 (2)	0.02	0.7	0.021	0.0063	0.014	32*
Arsenic	2.2	<0.0035	0.001	2.8	<0.0035	0.00074	0.000018	5.2
Barium	178	2.4 (2)	0.92	49.6	0.12	1.1	1	440
Beryllium	1.2	<0.00024	<0.002	0.5	<0.002	0.031	NA	160*
Boron	30.0	1.7	0.61	24.5	0.45	1.4	NA	7,200
Cadmium	0.6	<0.0013	<0.005			0.0078	0.0022	4.3
Calcium	28,500	1,330	1,230	1,500	14.0	NA	NA	NA
Chromium	777	<0.05	0.002	499	<0.005	23	0.74	120,000*
Chromium, +6	<0.40	NA	<0.02	<0.4	<0.02 (3)	0.047	0.011	37
Cobalt	44.5	0.43	<0.005	7.0	<0.005	0.94	NA	4,800*
Copper	28.5	0.014 (2)	0.003	15.8	0.003	1.3	0.0090	17
Iron	91,600	348	0.18	63,200	2.2	5	1	430,000
Lead	309	0.03 (2)	0.003	42.4	0.002 (2)	0.015	0.0025	400*
Magnesium	3,140	61.3	33.4	769	8.0	NA	NA	NA
Manganese	10,600	252	16.3	2,890	1.5	0.73	0.05	3,800* (4)
Mercury	<0.1	<0.002	<0.0002	0.2	<0.0002	0.0047	0.00005	0.06
Molybdenum	7.4	0.026 (2)	0.005	4.5	0.006	0.078	NA	400*
Nickel	91.8	0.5	<0.005	59.8	0.007	0.31	0.052	1,600*
Selenium	<0.5	<0.5	<0.005	0.5	<0.005	0.078	0.0050	400*

Table 3.27 - Initial Screening Analysis for Non-Bevill-exempt Nonwastewaters from the Chloride-Ilmenite Process

Constituent	DPE-SO-01 Total (mg/kg)	DPE-SO-01 TCLP (mg/L)	DPE-SO-01 SPLP (mg/L)	DPN-SO-01 Total (mg/kg)	DPN-SO-01 SPLP (mg/L)	HBL (mg/L)	AWQC (mg/L)	SSL (1) (mg/kg)
Silver	<0.1	<0.1	<0.001	0.2	<0.001	0.078	0.0034	400*
Thallium	3.7	0.28	0.012	7.2	<0.00225	0.0013	0.0017	6.4*
Tin	53.2	0.025	<0.01	12.9	<0.01	9.4	NA	48,000*
Titanium	6,380	<0.05	<0.005	5,360	<0.005	NA	NA	NA
Vanadium	240	0.0003 (2)	<0.005	1,060	<0.005	0.14	NA	720*
Zinc	122	1.1 (2)	0.03	57.2	0.073 (2)	4.7	0.12	24,000*
Dioxins/Furans (ng/kg; ng/L) (5)								
2378-TCDF (TEF=0.1)	12.2	NA	<0.010	121	<0.010	NA	NA	NA
Total TCDF	88.8	NA	<0.010	506	<0.010	NA	NA	NA
2378-TCDD (TEF=1)	<0.4	NA	<0.010	<0.3	<0.010	NA	NA	NA
Total TCDD	<0.4	NA	<0.010	<0.3	<0.010	NA	NA	NA
12378PeCDF (0.05)	21.8	NA	<0.051	371	<0.052	NA	NA	NA
23478PeCDF (0.5)	48.1	NA	<0.051	91.0	<0.052	NA	NA	NA
Total PeCDF	141	NA	<0.051	1,100	<0.052	NA	NA	NA
12378-PeCDD (TEF=1)	<1.8	NA	<0.051	<1.7	<0.052	NA	NA	NA
Total PeCDD	<1.8	NA	<0.051	<1.7	<0.052	NA	NA	NA
123478HxCDF (0.1)	237	NA	<0.051	2,490	<0.052	NA	NA	NA
123678HxCDF (0.1)	8.1	NA	<0.051	155	<0.052	NA	NA	NA
234678HxCDF (0.1)	2.5	NA	<0.051	74.1	<0.052	NA	NA	NA
123789HxCDF (0.1)	5.6	NA	<0.051	142	<0.052	NA	NA	NA

Table 3.27 - Initial Screening Analysis for Non-Bevill-exempt Nonwastewaters from the Chloride-Ilmenite Process

Constituent	DPE-SO-01 Total (mg/kg)	DPE-SO-01 TCLP (mg/L)	DPE-SO-01 SPLP (mg/L)	DPN-SO-01 Total (mg/kg)	DPN-SO-01 SPLP (mg/L)	HBL (mg/L)	AWQC (mg/L)	SSL (1) (mg/kg)
Total HxCDF	289	NA	<0.051	3,370	<0.052	NA	NA	NA
123478-HxCDD (0.1)	<1.8	NA	<0.051	<1.7	<0.052	NA	NA	NA
123678-HxCDD (0.1)	<1.8	NA	<0.051	<1.7	<0.052	NA	NA	NA
123789-HxCDD (0.1)	<1.8	NA	<0.051	<1.7	<0.052	NA	NA	NA
Total HxCDD	<1.8	NA	<0.051	2.8	<0.052	NA	NA	NA
1234678HpCDF (0.01)	189	NA	<0.051	1,520	<0.052	NA	NA	NA
1234789HpCDF (0.01)	126	NA	<0.051	1,690	<0.052	NA	NA	NA
Total HpCDF	366	NA	<0.051	3,710	<0.052	NA	NA	NA
1234678-HpCDD (0.01)	<1.8	NA	<0.051	<11.1	<0.052	NA	NA	NA
Total HpCDD	<1.8	NA	<0.051	15.8	<0.052	NA	NA	NA
OCDF (0.0001)	24,000	NA	<0.100	60,700	<0.100	NA	NA	NA
OCDD (0.0001)	22.2	NA	<0.100	404	<0.100	NA	NA	NA
2378 TCDD TEQ (5)	58.7 ppt	NA	ND	402 ppt	ND	0.0071 ppt	0.0031 ppt	45 (6)

NA - not available; ND - non detect

(1) Soil screening levels (SSLs) are based on soil background, except where soil ingestion levels are noted by the symbol “*”; in all cases the background levels are equivalent below soil ingestion levels. Air characteristic levels are noted when soil ingestion levels are exceeded. See “*Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes* (August 2000) in the docket for details and sources of the HBLs and SSLs.

(2) Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.

(3) Determined from DI water leach.

(4) The air characteristic level is 3,000 mg/kg at 25m and drops to 30,000 at 150m.

(5) TEQs calculated using WHO-TEFs.

(6) No air characteristic level has been determined.

DuPont Edge Moor, DE Facility

As described above, this facility markets a material that has been used as Subtitle D landfill cover, structural fill, and containment dikes for dredged spoils that is in part derived from non-Bevill-exempt solids (i.e., the filter solids from the production of ferric chloride and their wastewater treatment solids). The non-Bevill-exempt portion of the Iron Rich® is approximately 10% of the total volume of this waste. The entire volume of material was assessed in light of its current commingled status, the uncertainty associated with the actual percentage of non-Bevill-exempt material in the Iron-Rich®, and the physical impossibility of collecting a sample of the non-Bevill-exempt-only portion of this material. Sample DPE-SO-01 was used to assess the Edge Moor facility’s management practices. The SPLP constituents of concern exceeding the HBLs include antimony, arsenic, manganese, and thallium. Constituents exceeding the AWQC include antimony, arsenic, lead, manganese, and thallium. The TCLP constituents of concern include antimony, arsenic, barium, boron, iron, lead, manganese, and thallium.

EPA assessed the risks for disposal in an off-site industrial D landfill because this seemed to best fit the varied potential disposal or land-based use scenarios. The municipal landfill scenario may be less likely, but may still be relevant given the demonstration project at the nearby municipal landfill. Our assessment addresses the municipal scenario qualitatively. These scenarios were assessed for potential releases to drinking water wells. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the full groundwater pathway risk assessment.

We also conducted a screening analysis of the air pathway given the reported use of this material as landfill cover and caps and in other non-encapsulated methods. **Table 3.28** compares the constituents that were detected in the total analyses at levels exceeding the background soil levels to the soil ingestion HBLs and the Air Characteristics Study.

Constituent	Total Waste Concentration (DPE-SO-01) (mg/kg)	SSL (mg/kg)		
		Background Soil Level	Soil Ingestion HBL	Air Characteristics Study*
Antimony	0.9	0.7	32	NA
Beryllium	1.2	0.9	160	9,000 @25 m (adult)
Chromium	777	54	120,000	NA
Cobalt	44.5	9.1	4,800	3,000
Copper	28.5	25	NA	NA
Lead	309	19	400	600,000
Manganese	10,600	550	3,800	20,000 (@25 m) 100,000 (@150 m)

Table 3.28 - Comparison of Iron Rich® Total Analyses to Soil Screening Levels (SSL)

Constituent	Total Waste Concentration (DPE-SO-01) (mg/kg)	SSL (mg/kg)		
		Background Soil Level	Soil Ingestion HBL	Air Characteristics Study*
Molybdenum	7.4	1.0	NA	NA
Nickel	91.8	19	1,600	90,000
Thallium	3.7	1.9	6.4	NA
Vanadium	240	80	720	20,000
Zinc	122	60	24,000	NA
2378 TCDD TEQ	58.7 ppt	5 ppt	45 ppt	NA

*U.S. Environmental Protection Agency, "Revised Risk Assessment for the Air Characteristic Study", EPA 530-R-99-019a, November 1999, Table 4.1 (Landfills).
 NA = not available

This analysis shows that most metals present in the waste are at concentrations below their respective soil ingestion levels. The limited number of constituents exceeding the soil ingestion levels are still below the air characteristics study levels, indicating a minimal potential risk from the air pathway for a landfill scenario. However, we cannot judge with any certainty potential risks if the material were more widely dispersed, such as might occur in some of the projected uses reported by the generator.

New Johnsonville, TN Facility

The scenario of concern for this waste is the on-landfill and the Hillside Ponds. The SPLP results for Sample DPN-SO-01 (from the Hillside Pond) were used to screen the landfill and Hillside Pond scenario. The constituents of concern in the SPLP extracts were antimony and manganese. Arsenic and thallium are present in the wastes but are below the reported SPLP detection limits. Since ½ the SPLP detection limits is greater than the HBL for these two constituents, they were identified as potential constituents of concern. Considering AWQC, iron is a constituent of concern. Mercury also falls in this category (½ detection limit is above AWQC). Manganese also exceeds AWQC. The on-site landfill scenario was assessed for potential releases to surface water. We assessed whether the landfill or Hillside Ponds could impact groundwater that serves as a source of drinking water. As illustrated on the map provided by the facility in their RCRA §3007 Survey, the facility is located on a large tract of land bordered to the west by the Tennessee River. The management units are located on a ridge facing the river. Groundwater flow, while not

definitively studied, is expected to be toward the river.^{29,30} The facility did identify a drinking water well on the southeast corner of the plant property which is approximately 2 miles to the east of the management units. The groundwater pathway, therefore, did not seem plausible. Note, however, that the drinking water risks from releases to groundwater were assessed using the off-site landfill scenario noted above for the Delaware facility. Because any groundwater releases from these management units are likely to be toward the river, we assessed potential exposure to contaminated surface water for this site. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the full risk assessment.

DuPont DeLisle Plant; Pass Christian, MS Facility

As described above, this facility places its commingled wastewaters in on-site surface impoundments; the dredged solids from these units are placed in an on-site landfill. The solids filtered from this facility's ferric chloride are also placed in this landfill. No sample was collected from this site for this waste. However, EPA believes the sampling and assessment of the Tennessee and Delaware facilities is an appropriate surrogate for this waste given the similar nature of the processes.

3.2.10 HCl from Reaction Scrubber, Chloride-Ilmenite Process

This waste is generated by all three facilities that use the chloride-ilmenite process to produce titanium dioxide. This waste is generated as a result of the scrubbing of reactor off-gas and is recycled back to the process at the DuPont facilities in Tennessee and Mississippi. The DuPont facility in Delaware commingles this waste with other process wastewaters at the facilities wastewater treatment system. The waste from this facility is assessed below as part of the "Commingled wastewaters from chloride-ilmenite process".

3.2.11 Commingled Wastewaters from Chloride-Ilmenite Process

Waste Generation

Process wastewaters are generated at various points of the chloride-ilmenite production process. These wastewaters are generated from coke and ore solids recovery, reaction and oxidation scrubbers, spent caustic treatment, product finishing, raw material storage vent scrubbers, equipment vents and supernatant from wastewater impoundments, etc.

Waste Management Practices

At the DuPont facility in Delaware, the commingled wastewaters are treated in tanks and pass through an unlined cooling pond just before discharge via an NPDES permit. Discharge is through an unlined channel. The DuPont facilities in Tennessee and Mississippi manage their commingled

²⁹E-mail from Scott L. Goodman, DuPont to Max Diaz, U.S. EPA; December 9, 1999; RE: Drinking Water Well Location at New Johnsonville, TN Facility.

³⁰E-mail from Scott L. Goodman, DuPont to Max Diaz, U.S. EPA; March 22, 2000; RE: Groundwater Flow Direction at New Johnsonville, TN Facility.

wastewaters in a series of surface impoundments prior to NPDES discharge. The treated effluents from the wastewater treatment systems at these facilities are regulated under the Clean Water Act and were not assessed. However, the management of these commingled wastewaters in surface impoundments prior to discharge was evaluated. **Table 3.29** presents the reported volumes for the wastewaters that are commingled in the wastewater treatment systems at each facility.

Facility	Wastewater (RIN #)	Total Volume (MT/yr)
E.I. DuPont de Nemours & Co.; Edge Moor, DE	Reaction area & chlorine scrubber pretreated effluent (RIN 1)	10,521
	Coke & ore wash water (RIN 2)	97,611
	HCl pretreatment effluent (RIN 3)	169,609
	Chlorine scrubber water to WWT (RIN 7)	4,354
	SCP1 CaCl ₃ filtrate to WWT (RIN 8)	605,513
	Oxidation wastewater to reaction area pretreatment (RIN 9)	59,589
	Oxidation wastewater washwater (RIN 11)	5
	Finishing sump (RIN 10)	(Combined in RIN 11 volume)
	Purification wastewater (RIN 12)	1,769,305
	Dryer scrubber to WWT (RIN 13)	124,259
E.I. DuPont de Nemours & Co.; New Johnsonville, TN	Reaction scrubber pretreater wastewater (RIN 2)	246,000
	Purification scrubber wastewater (RIN 3)	82,000
	Solids removal wastewater (RIN 4)	442,800
	HCl storage tank scrubber (RIN 5)	9,110
	Finishing wastewater (RINs 6, 111, & 113)	6,566,998
	Chlorine unloading scrubber wastewater (RIN 101)	82
	Reaction startup scrubber (RIN 102)	3,268
	Reaction area maintenance scrubber (RIN 103)	11,112
	Oxidation scrubber (RIN 104)	6
	Maintenance wash house scrubber (RIN 105)	4540
	Hillside Pond wastewater (RIN 106)	102,200
	Reaction maintenance wastewater (RIN 109)	1,900

Table 3.29 - Volumes for Commingled Wastewaters from Chloride-Ilmenite Process

Facility	Wastewater (RIN #)	Total Volume (MT/yr)
	Wash house wash water (RIN 114)	454
E.I. DuPont de Nemours & Co.; DeLisle Plant; Pass Christian, MS	Scrubber wastewater (RIN 2)	216,286
	Reaction area scrubber pretreater wastewater (RIN 3)	328,802
	Wastewater from oxidation scrubber to spent chemical pretreatment (see RIN 7) (RIN 6)	9,259
	Spent chemical treatment wastewater (RIN 7) (treatment of RINs 6, 101, & 102)	24,997
	HCl storage vent scrubber (RIN 9)	3,835
	Chlorine unloading scrubber to spent chemical pretreatment (see RIN 7) (RIN 101)	202
	Emergency chlorine scrubber to spent chemical pretreatment (see RIN 7) (RIN 102)	15,536
	Scrubs unloading vent scrubber (RIN 104), finishing wastewater (RIN 8), & oxidation scrubber wastewater (RIN 103)	2,859,842
	Borrow Pit slurry water return to WWT (RIN 109)	191,000

Waste Characterization

Two samples were collected during record sampling to characterize this waste. At the DuPont facility in Delaware Sample DPE-WW-01 was collected directly from the influent to the finishing (cooling) pond. At the DuPont facility in Tennessee, Sample DPN-WW-01 was collected at the point where the wastewater leaves the weir and enters the first settling pond.

Totals analysis were conducted on Samples DPE-WW-01 and DPN-WW-01. A summary of the analytical results for each sample is presented in Appendix A. The detailed reports of the record sampling trips, the complete set of analytical data, and the validation reports for these samples are available in the “Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes From the Inorganic Titanium Dioxide Manufacturing Sector” for the DuPont Edge Moor, DE and New Johnsonville, TN facilities. These reports are available in the docket for this rulemaking.

Results of Screening Analysis

Table 3.30 presents the constituents detected in Samples DPE-WW-01 and DPN-WW-01 and the

associated HBLs and AWQC for each constituent.

Table 3.30 - Initial Screening Analysis for Commingled Wastewaters from Chloride-Ilmenite Process

Constituent	DuPont Edge Moor DPE-WW-01 (mg/L)	DuPont New Johnsonville DPN-WW-01 (mg/L)	HBL (mg/L)	AWQC (mg/L) (freshwater/saltwater)
Aluminum	0.65	3.1	16	0.087
Barium	0.62	0.030	1.1	1
Boron	0.72 B	0.05	1.4	NA
Chromium	<0.005	0.25	23	0.74
Chromium, +6	<0.02	<0.02*	0.047	0.011
Copper	0.03	0.007	1.3	0.009 /0.0031
Iron	1.44	16.7	5	1
Lead	<0.003	0.005 B	0.015	0.0025
Magnesium	142	5.19	NA	NA
Manganese	3.3	3.34	0.73	0.05
Molybdenum	0.009	0.006	0.078	NA
Nickel**	0.013	0.020	0.31	0.052 /0.0082
Thallium	<0.005	0.013	0.0013	0.0017
Titanium	0.32	13.6	NA	NA
Vanadium	0.018	0.63	0.14	NA
Dioxins/Furans (ng/L) *				
2378-TCDF (TEF=0.1)	<0.0096 ng/L	<0.010	NA	NA
Total TCDF	<0.0096	<0.010	NA	NA
12378-PeCDF (0.05)	<0.048	<0.052	NA	NA
23478-PeCDF (0.5)	<0.048	<0.052	NA	NA
Total PeCDF	<0.048	<0.052	NA	NA
123478-HxCDF (0.1)	<0.048	<0.052	NA	NA
123678-HxCDF (0.1)	<0.048	<0.052	NA	NA
234678-HxCDF (0.1)	<0.048	<0.052	NA	NA

Table 3.30 - Initial Screening Analysis for Commingled Wastewaters from Chloride-Ilmenite Process

Constituent	DuPont Edge Moor DPE-WW-01 (mg/L)	DuPont New Johnsonville DPN-WW-01 (mg/L)	HBL (mg/L)	AWQC (mg/L) (freshwater/ saltwater)
123789-HxCDF (0.1)	<0.048	<0.052	NA	NA
Total HxCDF	<0.048	<0.052	NA	NA
1234678-HpCDF (0.01)	<0.048	0.064 ng/L	NA	NA
1234789-HpCDF (0.01)	<0.048	<0.052	NA	NA
Total HpCDF	<0.048	0.064	NA	NA
Total HpCDD	<0.048	<0.052	NA	NA
OCDF (0.0001)	1.1 ng/L EB	1.4 ng/L	NA	NA
OCDD (0.0001)	<0.096	<0.10	NA	NA
2378-TCDD TEQ*	0.00011 ng/L or 0.11 ppq	0.00078 ng/L or 0.78 ppq	0.0071 ng/L	0.0031ng/L

NA-Not Available

*TEQs calculated using WHO-TEFs.

**Both of these values exceed the AWQC for saltwater. We will use the higher of the two values for DeLisle.

EB: Detected in equipment blank at 0.47 ng/L.

DuPont Edge Moor, DE

As discussed above, this facility treats wastewaters in tanks until just before discharge, when they pass through an unlined cooling pond. This unit is located on the north end of the plant adjacent to the Delaware River. The relevant sample of this wastewater was collected at the influent to the cooling pond, directly from the effluent clarifiers (DPE-WW-01). The constituents detected above the AWQC were aluminum, copper, iron, manganese, and nickel. Manganese was also detected above the HBL. It was assumed that any releases from the cooling pond at this facility would be intercepted by the river, and would be comparable in concentration, but much less volume than the actual NPDES discharge point which is subject to NPDES permitting.

DuPont New Johnsonville, TN

At this facility, the combined wastewaters are settled in a series of three impoundments prior to NPDES discharge. The relevant sample of the combined wastewater was collected prior to the settling ponds (DPN-WW-01) The constituents detected above the AWQC were aluminum, iron, lead, manganese, and thallium. Vanadium and thallium were also detected above the HBL.

The groundwater ingestion pathway is not believed to be of concern for this site as described in

the previous waste category. Thallium and vanadium, therefore, were not considered further for this facility. The ponds are adjacent to the Tennessee River. The surface impoundment scenario was assessed for infiltration and dilution to the Tennessee River. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the risk assessment.

DuPont DeLisle Plant; Pass Christian, MS

No samples were collected at this facility. As indicated above, this facility manages the commingled wastewaters in a series of three impoundments. We assessed this site to determine whether groundwater releases might impact drinking water wells on the vicinity. We obtained a USGS water well inventory printout for the 2 mile radius around the DeLisle plant.³¹ We also reviewed a USGS topographic map for the vicinity (see Appendix C). From these sources we determined that there are residences (a community named “Shell Beach”) and a “home” drinking water well approximately 2,000 feet to the south and west of the facility. There are additional residences to the west and numerous drinking water wells throughout the region. Given the hydraulic gradient depicted in the potentiometric map submitted in DeLisle’s RCRA §3007 Survey, the nearest drinking water well appears to be upgradient. It is not possible to tell definitively whether the other wells in the vicinity are down-gradient or side-gradient. The groundwater gradients may change offsite or change seasonally. We do not have sufficient information to rule out such possibilities. As a result, we modeled the potential impact of the impoundments on drinking water wells within a range of 2,000 to 3,000 ft. We also assessed the surface water pathway given the proximity of the impoundments to the Bay of St. Louis (located directly south of the facility). The analytical data from the Delaware and Tennessee facilities were used for the risk assessment. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the full risk assessment.

3.2.12 Aluminum-containing Additive Vent Filters Solids from Chloride-Ilmenite Process

The DuPont facility in Mississippi facility reported generating <1 MT/year of this waste. This waste is from vent filters used in the air pollution control devices for the oxidation process. This material is placed in an off-site industrial D landfill. Information from the facility indicates that this waste is predominantly composed of aluminum and is a small volume. The Agency, therefore, does not believe this material poses a risk to human health and the environment if it is disposed of in compliance with applicable Federal and state regulations.

3.2.13 Off-specification Titanium Dioxide Product

Waste Generation and Management

The DuPont facilities in Tennessee and Mississippi reported generating off-specification titanium dioxide product when the product specifications are not met and the material cannot be reworked

³¹ See Appendix C for facsimile from Heather Lott (USGS, Water Resources Division) to Max Diaz (EPA); Wells within a 2-Mile Radius of a Site in Pass Christian; August 29, 2000.

back into the process. The Tennessee facility indicated that this material usually is recycled back to the process, but the waste is sent to an off-site landfill when purity standards are not met. The Mississippi facility stores this waste in containers and then sends it an off-site landfill. **Table 3.31** presents a summary of the volumes for this waste.

Table 3.31 - Volumes of Off-specification Titanium Dioxide Product	
Facility (RIN #)	Total Volume (MT/yr)
E.I. DuPont de Nemours & Co.; New Johnsonville, TN (RIN 110)	295
E.I. DuPont de Nemours & Co.; DeLisle Plant; Pass Christian, MS (RIN 107)	267.8

Waste Characterization

A sample of this waste was collected from the Tennessee facility to characterize this waste. Sample (DPN-SO-02) was collected from a 50 pound bag located in the production warehouse.

Totals, TCLP and SPLP analyses were conducted on the sample. A summary of the analytical results for this sample is presented in Appendix A. The detailed report for the record sampling trip, the complete set of analytical data and the validation report for this sample is available in the “Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes From the Inorganic Titanium Dioxide Manufacturing Sector” for the DuPont New Johnsonville, TN facility. This report is available in the docket for this rulemaking.

Results of Screening Analysis

The landfills that accept this waste accept both municipal and industrial waste. Therefore, the TCLP (with somewhat higher leaching levels than the SPLP) results for Sample DPN-SO-02 were used to screen the municipal landfill scenario. **Table 3.32** presents the constituent(s) detected in Sample DPN-SO-02 and the corresponding HBLs.

Lead was the only constituent detected in the TCLP above the HBL and AWQC. Boron also exceeded the HBL, but only by a factor of 1.5. This constituent was therefore screened out. The off-site municipal landfill scenario was assessed for lead. Refer to the *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes (August 2000)* for the details of the full risk assessment.

Table 3.32 - Initial Screening Analysis for Off-specification Titanium Dioxide Product		
Constituent	DPN-SO-02 TCLP (mg/L)	HBL (mg/L)
Aluminum	60.8	16
Antimony	0.007 *	0.0063
Barium	<2	1.1

Table 3.32 - Initial Screening Analysis for Off-specification Titanium Dioxide Product

Constituent	DPN-SO-02 TCLP (mg/L)	HBL (mg/L)
Boron	2.2	1.4
Chromium	<0.05	23
Cr,+6	NA	0.047
Copper	<0.25	1.3
Iron	<1	5
Lead	0.06*	0.015
Magnesium	1.0	NA
Nickel	<0.2	0.31
Tin	<0.5	9.4
Titanium	0.18	NA
Zinc	0.88*	4.7

NA- Not Available

*Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.

** Determined from DI water leach.

3.2.14 Railcar/Trailer Product Washout

The DuPont facility in Tennessee reported generating this waste when the tank cars and railcars used to ship the TiO₂ to customers are cleaned. The washwater, containing titanium dioxide, is placed in a surface impoundment. The water from this pond is subsequently sent to wastewater treatment where it is commingled with all other chloride-ilmenite wastewaters, (discussed above in Section 3.2.11). The titanium dioxide product that settles to the bottom of this pond is mechanically removed and reused in the production process. The potential impact of this impoundment was screened using the SPLP analysis collected for Sample DPN-SO-02 of off-specification product. The SPLP results were used to assess this management scenario because there is no potential for contact with municipal landfill leachate. There were no constituents concern detected above their HBLs in the SPLP analysis of this sample. This waste did not warrant further assessment.

Table 3.33 - Initial Screening Analysis for Railcar/Trailer Product Washout

Constituent	DPN-SO-02 SPLP (mg/L)	HBL (mg/L)
Aluminum	0.05*	16
Antimony	0.008	0.0063

Table 3.33 - Initial Screening Analysis for Railcar/Trailer Product Washout

Constituent	DPN-SO-02 SPLP (mg/L)	HBL (mg/L)
Barium	0.054	1.1
Boron	0.17	1.4
Chromium	0.001*	23
Cr,+6	<0.02**	0.047
Copper	0.002	1.3
Iron	<0.05	5
Lead	0.002*	0.015
Magnesium	0.08	NA
Nickel	<0.005	0.31
Tin	<0.01	9.4
Titanium	<0.005	NA
Zinc	0.02*	4.7

NA- Not Available

*Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.

** Determined from DI water leach.

3.3 OUT OF SCOPE WASTE

In addition to the wastes presented in Tables 3.1, 3.2 and 3.3, there are other wastes generated during the production of titanium dioxide that are beyond the scope of the consent decree.

3.3.1 Bevill-exempt Wastes

The consent decree does not require EPA to make listing determinations for wastes that are Bevill exempt under EPA rules implementing the so-called “Bevill exemption” for mining wastes. As stated in 40 CFR 261.4(b)(7)(ii)(S), chloride process waste solids from titanium tetrachloride production are Bevill-exempt waste. These solids are generated during the chlorination reaction of the titanium ore in the reducing presence of coke at elevated temperatures, and are generated from both the chloride process and the chloride-ilmenite process. The majority of these solids leave the reactor as a mass and are quenched, neutralized, settled and disposed as Bevill-exempt materials. Additional solids from the reactor are carried overhead with the TiCl₄ and are subsequently removed in various scrubbing units. These solids are also identified as Bevill-exempt solids as they are derived from the same unreacted ore and coke solids leaving the chlorination reactor. These Bevill-exempt waste solids that are generated at the facilities that use the chloride or the chloride-ilmenite process to produce titanium dioxide are discussed below.

Solids also are generated from the oxidation and finishing stages of titanium dioxide production that are captured in air pollution control devices such as scrubbers. These solids are non-Bevill-exempt solid wastes (not covered by the Bevill exemption). Most titanium dioxide producers commingle wastewaters from titanium tetrachloride production with wastewaters from oxidation and finishing, resulting in wastewater treatment sludges with Bevill-exempt and non-Bevill-exempt components.

Due to process variations, each facility generates its Bevill-exempt and non-Bevill-exempt solids in slightly different ways. The following is a discussion of the status, Bevill exempt vs non-Bevill exempt, of these solids at the facilities that use the chloride or chloride-ilmenite process.

Waste Solids from Titanium Tetrachloride Production via the Chloride Process

All of the facilities that use the chloride process generate these waste. These solids are generated as a result of the separation of the residual coke and ore from the titanium tetrachloride product stream produced during the chlorination process. As discussed above, these solids are Bevill exempt and are outside of the scope of the consent decree. However, some of these facilities commingle this Bevill-exempt waste with non-Bevill-exempt wastewaters in their on-site wastewater treatment systems.

Based on the information available to EPA, it appears that *Kemira* and *Louisiana Pigment* do not commingle these 100 percent Bevill-exempt waste solids from the production of titanium tetrachloride with any other waste. *Kemira* sends these solids to a dedicated settling pond and *Louisiana Pigment* uses a tank based system to segregate these solids and then sends the solids to an on-site landfill. At these two facilities, the solids from the production of titanium tetrachloride are clearly outside of the consent decree and do not warrant further assessment.

The *Kerr-McGee* facility commingles these solids with wastewaters from the production of sodium chlorate in the facility's wastewater treatment system. The sodium chlorate wastewaters account for a small percentage of the total volume of managed wastewater, and solids generated.³² It does not appear that *Kerr-McGee* commingles any wastewaters from oxidation or finishing (that might contain non-Bevill-exempt solids).

At both of the *Millennium* facilities in Ohio, the wastewaters from titanium tetrachloride production that bear the Bevill-exempt solids are commingled with wastewaters from oxidation and finishing. Although neither facility reported any solids in the oxidation or finishing wastewaters, data from similar wastewaters from the chloride-ilmenite process indicate that very low levels of solids are present in these wastewaters.

At *Kemira* and the *Millennium Baltimore* facility, chloride process waste solids also are collected in the wastewater treatment systems. Both facilities commingle sulfate and chloride process wastewaters in their wastewater treatment systems. The resulting wastewater treatment sludge is composed of a significant amount of non-Bevill-exempt solids. These wastewater treatment solids are discussed in **Section 3.2.7** as part of the "Wastewater Treatment Sludges

³² The analytical results for this sample can be found in the "*Sodium Chlorate Listing Background Document*." The predominant potential constituent of concern in the sodium chlorate solids is chromium; the analytical data for the commingled solids (KM-SI-04) show that the SPLP concentration is <0.05 mg/L and therefore not of concern.

From Commingled Chloride and Sulfate Process Wastewaters” waste category.

Table 3.34 presents the “Bevill-exempt” waste solids generated during the production of titanium tetrachloride at the facilities that use the chloride processes. The facilities were only required to identify Bevill-exempt wastes in the RCRA §3007 questionnaire. No volume or waste management information was required.

Table 3.34 - Bevill-exempt-Waste Solids from Titanium Tetrachloride Production Via the Chloride Process	
Facility	Waste (RIN #)
Kemira Pigments, Inc.	Chlorination waste solids (RIN 6)
Millennium Inorganic Chemicals, Baltimore, MD	Filter Cake (RIN 4) ³³
Kerr-McGee Chemicals Corporation	Chlorinator Solids (RIN 3) ³⁴
Kronos/Louisiana Pigment	Filter Cake (RIN 5)
Millennium Inorganic Chemicals, Plants I and II, Ashtabula, OH	Nonhazardous filter cake (RIN 4)

Solids from the Chloride-Ilmenite Process

All three chloride-ilmenite facilities generate waste solids from the chloride-ilmenite process. The DuPont facilities in Tennessee and Mississippi use landfills to manage these wastes. The DuPont facility in Delaware is currently managing this waste in a landfill, but has used the material for various land application purposes in the past. The solids managed in these landfills contain contributions from Bevill-exempt mineral processing solids as well as non-Bevill-exempt solids. The site-specific variations in the processes determined the composition of the Bevill-exempt versus the non-Bevill-exempt solids in these units. The generation and management of these Bevill-exempt and non-Bevill-exempt solids is discussed below for each facility.

DuPont Edge Moor, DE Facility

The DuPont facility in Delaware generates solids at three places in their production process. These solids include the Bevill-exempt chloride process solids from the titanium tetrachloride production process (reactor solids), solids removed from ferric chloride waste acid, and wastewater treatment sludge. A portion of these Bevill-exempt reactor solids (unreacted coke and ore) are recycled to the process. This facility combines and neutralizes these three sources of solids and markets this material as “Iron Rich®” for a variety of uses.

³³Generated in a surface impoundment of the facility’s wastewater treatment system. Assessed as part of “Wastewater Treatment Sludges from Commingled Chloride and Sulfate Process Wastewaters” waste category in **Section 3.2.7**.

³⁴The name and description for this waste is from telephone contact with the facility. It should also be noted that the facility did not report any wastewaters or solids from oxidation and finishing.

The Delaware facility adds a processing chemical prior to removal of solids from the ferric chloride to improve the ferric chloride properties. The production of ferric chloride is not mineral processing, and this process step is chemical manufacturing (and/or an ancillary operation) beginning at the point where the facility adds the processing chemical prior to solids removal. Since the solids are removed after the point in the process where mineral processing ends, they are non-Bevill-exempt solid wastes.

Per the data reported by the Delaware facility in their RCRA §3007 questionnaire, 8.3% of the wastewater treatment solids are derived from oxidation and finishing. The assessment of the solids generated at the Delaware facility is presented in Section 3.2.9 as part of the “Non-Bevill-exempt nonwastewaters from the Chloride-Ilmenite Process” waste category.

DuPont DeLisle Plant; Pass Christian, MS

The DuPont facility in Mississippi generates the bulk of its Bevill-exempt solids from the filtration of the ferric chloride waste acid and a lesser amount in the wastewater treatment system at the facility. The solids from the filtration of the ferric chloride are Bevill exempt because this step is simply removal of Bevill-exempt solids prior to disposal of the waste acid. The solids are placed in a pond, and the dredged solids from the pond are placed in a dedicated on-site landfill cell. The facility also reported three additional Bevill-exempt waste streams that were generated as a result of the recovery of coke and solids from the initial chlorination reaction. The solids generated in the facility’s wastewater treatment system are assessed as part of “Non-Bevill-exempt nonwastewaters from the chloride-ilmenite process” waste category, discussed in Section 3.2.9.

DuPont New Johnsonville, TN

The DuPont facility in Tennessee also generates the bulk of its Bevill-exempt chloride process solids from the filtration of the ferric chloride waste acid and a small amount in the wastewater treatment system at the facility. This facility removes the bulk of the unreacted coke and ore solids during the filtration of their ferric chloride. These solids are landfilled as a discrete waste. The wastewater treatment solids are discussed in Section 3.2.9 as part of the “Non-Bevill-exempt Nonwastewaters from the Chloride-Ilmenite Process” waste category.

Table 3.35 presents the “Bevill-exempt” waste solids generated during the production of titanium tetrachloride during the chloride-ilmenite process. The facilities were only required to identify Bevill-exempt wastes in the RCRA §3007 questionnaire. No information on the volumes generated or management practices were required.

Table 3.35 - Bevill-exempt Waste Solids from Titanium Tetrachloride Production via the Chloride-Ilmenite Process	
Facility	Waste Description (RIN #)
E.I. DuPont de Nemours & Co.; Edge Moor, DE	Coke and ore solids ³⁵

³⁵Facility did not assign RIN.

Table 3.35 - Bevill-exempt Waste Solids from Titanium Tetrachloride Production via the Chloride-Ilmenite Process	
Facility	Waste Description (RIN #)
E.I. DuPont de Nemours & Co.; DeLisle Plant; Pass Christian, MS	Reaction area solids (RIN 18)
	Solids recovery area silica (RIN 13)
	Solids recovery recovered cole & ore (RIN 14)
	Solids recovery silica (RIN 15)
	Environmental coke and ore solids (RIN 16)
E.I. DuPont de Nemours & Co; New Johnsonville, TN	Titanium tetrachloride solids ³⁶
	Coke and ore solids (RIN 9) ³⁷

In addition to the Bevill-exempt mineral processing wastes (chloride process waste solids), beneficiation wastes generated from the storage and handling of various raw materials are also Bevill exempt. The three chloride-ilmenite facilities generate several wastes as a result of the storage and handling of the process raw materials. Kronos/ Louisiana Pigments also reported generating filter cloths as a result of the dewatering of slurry from the chlorination process. These filter cloths are stored in roll-off bins because the facility has not located a landfill to take this waste because of elevated NORM (radium 226 and 228). **Table 3.36** presents the Bevill-exempt beneficiation waste associated with the storage and handling of the process raw materials.

Table 3.36 - Bevill-exempt Storage and Handling Wastes	
Facility	Waste Name (RIN #)
E.I. DuPont de Nemours & Co.; DeLisle Plant; Pass Christian, MS	Coke and ore storage and unloading bag dust (RIN 10)
	Ore dust (RIN 11)
	Transfer pump solids to landfill (RIN 12)
	Coke and ore storage dust (RIN 110)
	Coke and ore transfer dust collection to storage (RIN 111)
	Coke and ore transfer pump (RIN 112)
	Recycle water storage solids (RIN 113)
E.I. DuPont de Nemours & Co.; Edge Moor, DE	Dust control (RIN 21)
Kronos/Louisiana Pigment	Filter cloths (RIN 12)

³⁶Ibid.

³⁷Solids from filtration of iron chloride.

The DuPont facility in Mississippi reported two additional Bevill-exempt wastes (“Recycle water solids”- RIN 17 and “Solids with water”- RIN 18) that are recycled back to the process.

3.3.2 Debris and Non-Process Wastes

Some kinds of debris and plant component materials do not fall within the scope of the consent decree. Most of the wastes that fell in this category were refractory brick wastes generated when facilities refurbished plant furnaces. This material is derived from a structural component of the plant where production takes place rather than a waste from the “production” of an inorganic chemical. This debris from process equipment is also out of scope of this consent decree. **Table 3.37** presents the reported volumes for these materials.

Table 3.37 - Debris and Non-Process Waste			
Facility	Material (RIN #)	Management	Total Volume (MT/yr)
Kronos/Louisiana Pigment Co.	Chlorinator Bed Material (RIN 10)	On-site landfill	49
	Refractory Brick (RIN 9)	Off-site Subtitle C landfill	594
	Blasting Material (RIN 11) ³⁸	Off-site Subtitle C landfill	105

3.3.3 National Pollutant and Discharge System (NPDES)

Industrial wastewater discharges that are point sources and subject to regulation under Section 402 of the Clean Water Act, are not solid wastes subject to RCRA (see 40 CFR 261.4(a)(2)). Several of the titanium dioxide facilities reported discharging treated wastewaters to surface waters under the National Pollutant and Discharge Elimination System (NPDES). These discharges require a permit and are regulated under the Clean Water Act (Act) and were not evaluated. **Table 3.38** presents the reported volumes for these permitted discharges.

Table 3.38 - Permitted NPDES Waste		
Facility	Waste (RIN #)	Total Volume (MT/yr)
Kemira Pigments, Inc.	Gypsum plant effluent (RIN 12)	3,779,000
	Effluent from Wastewater Treatment System (RIN 11)	25,166,000
Millennium Inorganic Chemicals Baltimore, MD	Treated Effluent from sulfate digestion scrubber water (RIN 6)	11,720,000

³⁸Sand used to clean the walls of the chlorinator prior to replacement of refractory

Table 3.38 - Permitted NPDES Waste		
Facility	Waste (RIN #)	Total Volume (MT/yr)
	Effluent from Gypsum production (RIN 11)	1,240,988
	Effluent from Wastewater Treatment System (RIN 3)	2,961,801
Kerr-McGee Chemicals Corporation	Effluent from Wastewater Treatment System	6,879,798
Kronos/Louisiana Pigment Co.	Effluent from Wastewater Treatment System (RIN 6)	707,882
	Finishing Wastewater (RIN 7)	695,605
Millennium Inorganic Chemicals; Plant I Ashtabula, OH	Effluent from Wastewater Treatment System (RIN 3)	4,500,000
Millennium Inorganic Chemicals, Plant II; Ashtabula, OH	Effluent from Wastewater Treatment System (RIN 3)	5,500,000
E.I. DuPont de Nemours & Co. New Johnsonville, TN	Effluent from Wastewater Treatment System	Not Reported
E.I. DuPont de Nemours & Co.; DeLisle Plant; Pass Christian, MS		
E.I. DuPont de Nemours & Co.; Edge Moor, DE		

3.3 FORMATION OF DIOXINS/FURANS IN CHLORINATOR

In developing the sampling and analysis protocols for this sector, EPA determined that chlorinated dioxins and furans were potential constituents of concern. We were concerned about the potential presence of these compounds in this sector's wastes because the reaction conditions required to produce titanium tetrachloride from titanium ores appear to be similar to conditions at other processes known to be associated with dioxin/furan formation.

The initial reaction in the production of titanium dioxide is described in a DuPont patent as follows (emphasis added):

A reduction/chlorination process is provided for the treatment of titaniferous materials such as ilmenite ores. The chlorination is selective in that the titanium constituents of the titaniferous material is chlorinated, but there is no appreciable net yield of iron chloride from the iron constituent. Where other metals such as vanadium are present they may be chlorinated with the titanium. *The reduction utilizes as the reductant an amount of carbonaceous material which, based on oxygen in the titaniferous material, is at least stoichiometric to produce carbon monoxide. The selective chlorination utilizes as the*

chlorinating agent either ferrous chloride (FeCl₂) alone or certain combinations of ferrous chloride and one or more other chlorine-containing members, notably molecular chlorine (Cl₂) and hydrogen chloride (HCl). The use of ferric chloride (FeCl₃) as a part or all of the chlorinating agent is the equivalent of using a FeCl₂/0.5 Cl₂ mixture.

Preferably, sufficient chlorine atoms are provided by the chlorinating agent to react with essentially all of the titanium in the titaniferous material. An elevated temperature in the range of 950° to 1400°C is maintained during the chlorination, but depending upon the composition of the chlorinating agent, there may be a minimum temperature in the range which is needed to maintain selectivity.³⁹

The patent illustrates a number of conditions which are important for the formation of dioxins and furans: a carbon source, a chlorine source, and a heat source. The chloride process and the chloride ilmenite process utilize these conditions in their reactors to convert titanium ores and slags into titanium tetrachloride, which is subsequently purified and then oxidized to form the product titanium dioxide. EPA has catalogued many different sources of dioxins and furans, the vast majority of which involve these three critical conditions of carbon, chlorine and heat.^{40, 41}

The results of EPA's record sampling and analysis for wastes from the production of titanium dioxide confirmed the expectation that dioxins and furans were likely to be formed during the chlorination process. The following samples (with their respective TCDD TEQs) were collected from wastes generated in conjunction with the chlorinators:

- C Millennium Baltimore, MI-WW-01, Chloride solids/waste acid (RIN 1): 812 ng/L
- C Millennium Baltimore, MI-SO-01, Filter press solids (RIN 4)⁴²: 2,615 ng/kg.
- C DuPont Edge Moor, DPE-SO-01, Iron Rich: 58.7 ng/kg.
- C DuPont New Johnsonville, DPN-SO-01, Wastewater treatment solids: 402 ng/kg.

As described in earlier sections, each of these wastes contains significant levels of solids from the chlorinator. These chlorinator solids are classified as the exempt mineral processing waste from the production of titanium tetrachloride. Based on engineering judgement, we expect that dioxins and furans formed in the chlorinator would adhere to these solids given the affinity of these compounds to solids, as well as their low volatility and solubility.

[Chlorinated dioxins and furans] have a low solubility in water and a low volatility.

³⁹United States Patent. Number 3,977,863. August 31, 1976. "Process for Selectively Chlorinating the Titanium Content of Titaniferous Materials" Assignee: E.I. Du Pont de Nemours and Company, Wilmington, Delaware.

⁴⁰Cleverly, D., J. Schaum, D. Winters, G. Schweer. 1999. Inventory of sources and release of dioxin-like compounds in the United States. Presented at Dioxin '99, the 19th International Symposium on Halogenated Environmental Organic Pollutants and POPs, held September 12-17 in Venice, Italy. Short paper in, *Organohalogen Compounds*, Volume 41:467-472.

⁴¹Toxicological Profile for Chlorinated Dibenzo-p-dioxins (Update). U.S. Department of Health & Human Services. Public Health Service. Agency for Toxic Substances and Disease Registry. December 1998

⁴²This residual is generated from treatment of RIN 1 (Chloride solids/waste acid) and other commingled wastes.

Chlorinated dioxins have an affinity for particulates and readily partition to particles in air, water, and soil.⁴³

Our data support this contention that the dioxins and furans formed in the chlorinator would tend to adhere to the reactor solids:

- C The Millennium Baltimore chloride solids/waste acid (RIN 1, MI-WW-01) conveys the reactor solids to treatment and contained 812 ng/L 2,3,7,8-TCDD TEQ. The reactor scrubber water (RIN 2, MI-WW-02), in contrast, contained only 0.000018 ng/L 2,3,7,8-TCDD TEQ.
- C Kerr-McGee commingles all wastewaters and reactor solids in a series of impoundments. The total analysis of this commingled wastestream contained 65.6 ng/L 2,3,7,8-TCDD TEQ. In contrast, the SPLP filtrate portion of Kerr-McGee's wastewater (KM-SI-01) contained no detectable dioxins or furans.
- C Kemira's wastewater (KP-WW-01), sampled at the influent to their settling impoundment contained 0.000038 ng/L 2,3,7,8-TCDD TEQ.
- C DuPont Edgemoor's treated wastewater (DPE-WW-01) contained only 0.00011 ng/L 2,3,7,8-TCDD TEQ in contrast to their Iron Rich (DPE-SO-01) which contained 58.7 ng/kg 2,3,7,8-TCDD TEQ.
- C Similarly, DuPont New Johnsonville's wastewater (DPN-WW-01) contained only 0.00078 ng/L 2,3,7,8-TCDD TEQ, while their wastewater treatment solids (DPN-SO-01) contained 402 ng/kg 2,3,7,8-TCDD TEQ.

Our listing determination for K178 differentiates between exempt and non-exempt solids. With one exception⁴⁴, the non-exempt solids are associated with oxidation and finishing wastewaters. We do not believe that there should be dioxin/furan contamination of these non-exempt solids from oxidation and finishing. The oxidation step is designed to remove chlorine as the titanium tetrachloride is oxidized to form titanium dioxide. The chlorine offgas is returned to the chlorinator. In addition, at this point in the process we believe that very little carbon (one of the 3 important dioxin precursors) remains in the product stream as a result of the numerous purification steps which occur prior to oxidation. Our prediction that the dioxins and furans are not associated with the non-exempt solids from oxidation and finishing is borne out by the following analytical results:

- C A sample of off-specification titanium dioxide (DPN-SO-02) collected at DuPont New Johnsonville, contained 0.0026 ng/kg 2,3,7,8-TCDD TEQ.
- C The Kemira settling pond solids (KP-SO-02) which contained the highest percentage of non-exempt solids (>35 %) of the 5 facilities sampled contained the lowest level of 2,3,7,8-TCDD TEQ (3 ng/kg).

For these reasons, we conclude that the dioxins and furans detected in the record samples

⁴³Toxicological Profile for Chlorinated Dibenzo-p-Dioxins (update), p. 357.

⁴⁴As described in Section 3.2.9, DuPont Edge Moor removes solids from its ferric chloride waste acid. Comparable solids generated at DeLisle and New Johnsonville are exempt solids. Edge Moor's solids, however, lose their exempt status due to the processing that Edge Moor conducts prior to solids removal (i.e., mineral processing and the production of titanium tetrachloride have ended and the ferric chloride processing is considered chemical manufacturing and/or an ancillary operation).

supporting the titanium dioxide listing determinations are associated with the exempt solids, or with the ferric chloride solids,⁴⁵ and therefore are not within the scope of the consent decree. These compounds were not assessed as part of the rulemakings which established the mineral processing exemptions, and so these results could present new issues for these wastes if such compounds were found to pose unacceptable risks. During the development of the mineral processing exemption, EPA anticipated certain conditions might suggest the appropriateness of re-opening these exemptions⁴⁶. We are considering whether we should re-assess the status of these wastes as exempt mineral processing wastes. Any reassessment of these wastes would involve a separate analysis and opportunity for notice and comment.

⁴⁵ Ibid.

⁴⁶“If EPA finds that this exemption is not protective of human health and the environment and if an examination of titanium tetrachloride waste management shows any continuing or new problems, the Agency will reconsider this subtitle D determination for chloride process waste solids from titanium tetrachloride production.” 56 FR 27300, June 13, 1991.

APPENDIX A
SUMMARIES OF ANALYTICAL DATA RESULTS FOR EACH WASTE SAMPLE

APPENDIX B
SUMMARIES OF ANALYTICAL RESULTS FOR SPLIT-SAMPLES