United States
Environmental Protection
Agency

Office of
Solid Waste
Washington, D.C. 20460

October 2001

Response to Comments Document

Hazardous Waste Listing Determination for Inorganic Chemicals Industry Wastes (Final Rule)

Volume III:
Response to Comment ICMP-00022

Table of Contents

Public Comments on the
Inorganic Chemical Manufacturing Listing Determination
(Proposed September 14, 2000)

Comment ICMP-00022, E.I. du Pont de Nemours

Comment 22-1: DuPont Concurs with Several Positions Adopted by EPA in this Proposal.

Comment 22-1a: DuPont concurs with EPA's position that salt manufacturing is a separate production process outside the scope of the consent decree.

In Section 111.2, 65 FR 55688, EPA states that they considered salt-making to be a separate production process outside the scope of the consent decree and are not proposing any listings for wastes generated in the salt plant.

DuPont fully supports EPA's position that salt manufacturing is outside the scope of the 14 inorganic chemical sectors set out in the consent decree. Iron chloride solution generated at the DuPont Tennessee facility is used as a raw material ingredient in an industrial process to make a product. Prior to its use in salt manufacturing the iron chloride solution is managed in enclosed tanks and piping and poses no exposure pathway potential.

Response 22-1a: EPA acknowledges DuPont’s support.

Comment 22-1b: DuPont concurs with EPA's proposal not to list titanium dioxide from railcar/trailer product washout.

At 65 FR 55766, the Agency proposes to not list railcar/trailer product washout placed in a surface impoundment. We agree with the Agency that analytical results of this material would be similar to those obtained from the off-specification titanium dioxide and pose no risk. However, we would disagree with EPA's referring to this material as a waste. The Ti02 solids are recovered from this surface impoundment and returned back to the process as recovered product. The Agency states in the Land Disposal Restrictions Phase IV at 63 FR 28584 (May 26, 1998) "if wastes accumulate in piles or impoundments, if those wastes are hazardous .. . and the wastes are not legitimately recycled, then the units are Subtitle C regulated units because they are being used to store or dispose of hazardous waste." [emphasis added] Besides being nonhazardous, this material is legitimately recycled and is therefore not a waste.

Elsewhere in these comments we have described how the existing listing description would include these solids if disposed.

Response 22-1b: EPA acknowledges DuPont’s support. See comment 9-2c.

Comment 22-1c: DuPont concurs with EPA's decision not to list chloride-ilmenite process iron chloride ("waste acid") as hazardous waste.

The Agency has decided not to list iron chloride ("waste acid") from the chloride ilmenite process at 65 FR 55759-55760 and will rely on the hazardous characteristics of the material for any necessary control. DuPont supports the Agency's position.

All of these iron chloride streams are sold as products, used as ingredients or managed in permitted underground injection wells with approved no-migration petitions.

At the Delaware facility, ferric chloride is a commercial product and is sold to a leading North American coagulant manufacturer and marketer for resale into commercial potable water markets, municipal wastewater treatment markets, and industrial wastewater treatment applications. It has been sold commercially since 1974, prior to enactment of the Resource Conservation and Recovery Act of 1976. This material meets DuPont product specifications as well as meeting specifications established by NSF that are based on standards established by the American Water Works Association and followed by its members. The legitimacy of this product is discussed in detail in Section 11.0 of DuPont's comments [see comment 22-11].

At the Tennessee facility, iron chloride material is used as an ingredient in an industrial process to make a product. DuPont concurs with the Agency's statement at 65 FR 55760 that there are no known exposure routes associated with management of the material prior to inserting it into the non-consent decree production process.

At the Mississippi facility, the iron chloride solution is managed as a RCRA hazardous waste in accordance with full Subtitle C standards and disposed in underground injection wells with an approved no-migration petition. Prior to injection, the waste is managed in treatment and storage tanks that meet full RCRA Subtitle C standards; one of these tanks operates under a RCRA storage permit issued by the Mississippi Department of Environmental Quality (MDEQ). The injection wells are operated under a permit issued by the MDEQ as Class I hazardous waste injection wells. A no-migration petition was submitted to EPA when land disposal restrictions were imposed on the waste; it was approved in May 2000. The petition grants an exemption to land disposal restrictions because the Regional Administrator has determined that the method of disposal is protective of human health and the environment. A method of land disposal may not be determined to be protective unless "it has been demonstrated to the Administrator to a reasonable degree of certainty, that there will be no migration of hazardous constituents from the disposal unit or injection zone for as long as the wastes remain hazardous" [45 U.S.C. § 6924(g)(5)].

Although the Agency specifically proposes not to list iron chloride solution, the derived from rule could attach the K178 listing code to the stream. At the Mississippi facility, the iron chloride stream is generated with the Bevill-exempt coke and ore solids and a "nonexempt" portion derived from the vanadium waste. The stream as generated contains a nominal 4% solids by weight, and thus would carry the nonwastewater designation under the land disposal restrictions. After removal of the solids, the stream as injected is a wastewater with a very low solids content. The derived-from rule could be interpreted to result in the deepwell injected stream to carry the K178 waste code. The Agency should craft the final rule so that the iron chloride stream clearly does not carry the K178 listing code and the derived-from rule does not attach the listed code to this stream.

Response 22-1c: EPA acknowledges DuPont's support for its decision not to list chloride-ilmenite process iron chloride ("waste acid") as hazardous waste. As finalized, the K178 listing has no impact on the Mississippi facility because this plant is not manufacturing ferric chloride acid for subsequent sale or use. Thus, it does not generate any solids from the manufacture of ferric chloride. In addition, the Agency is not finalizing the portion of the proposal that would have incorporated solids derived from the vanadium stream within the scope of the listing.

Comment 22-1-d: DuPont concurs with EPA's position that chloride-ilmenite coke and ore solids are covered by the Bevill exemption

EPA discusses solids generated in the chloride-ilmenite process at 65 FR 55760, and coke and ore solids are identified as Bevill-exempt solids from the production of titanium tetrachloride. DuPont supports this conclusion. These solids are composed of nonvolatile chlorides and unreacted ore and coke from the chlorination reactor and clearly fit within the plain language of the exemption.

Although the Agency did not evaluate wastes that maintain the Bevill exemption, the effect of the proposed rule as written is that these wastes are included in the K178 listing. The Agency states that while the chloride-ilmenite coke and ore solids streams are largely exempt, any contribution to the stream from the vanadium waste ("in-process titanium tetrachloride purification stream") is non-exempt. As discussed in Section 3 of these comments [see comment 22-3], DuPont disagrees that the presence of the solids derived from the "in-process titanium tetrachloride purification stream" nullifies the Bevill exemption for the chloride process waste solids and causes the entire stream to be a listed hazardous waste.

EPA's position that the coke and ore solids that are removed from the gaseous titanium tetrachloride product stream, quenched and neutralized are "largely exempt" is consistent with the Agency's determination in its Bevill determinations of the late 1980s and early 1990s.

In EPA's April 17, 1989 proposal, it identified as "conditionally retained within the Bevill exclusion" certain mineral processing wastes meeting its proposed definition, including "chloride processing waste solids from titanium and titanium dioxide production." (54 FR 15317, 15323). In the September 1, 1989 final rule, EPA retained as part of the 33 wastes proposed to be conditionally retained within the Bevill exclusion "chloride processing waste solids from titanium and titanium dioxide production." (54 FR 36593)

In the September 25, 1989 proposed rule, the Agency proposed to continue to retain as Bevill exempt "chloride process waste solids from titanium tetrachloride production." (54 FR 39298)

In the January 23, 1990 final rule, EPA decided to remove five (5) of the twenty (20) mineral processing wastes previously proposed for Bevill exempt status. The fifteen (15) remaining streams designated at Bevill exempt included the "chloride process waste solids from titanium tetrachloride production" (55 FR 2323). In this final rule, the Agency concluded that "all solid wastes generated by this process (chloride-ilmenite process) are subject to EPA's reinterpretation of the Mining Waste Exclusion, including this rulemaking." (55 FR 2329) Thus, EPA considered that "all solid wastes" generated by DuPont's chloride-ilmenite process were "subject to the EPA's reinterpretation of the Mining Waste Exclusion" or in other words were considered to be Bevill exempt, and not regulated under RCRA. These "solid wastes" from DuPont's chloride-ilmenite plants included the "coke and ore solids" resulting from the production of titanium tetrachloride. These are the same solids EPA continues to recognize in the September 14, 2000 proposal are Bevill exempt, as long as they are not commingled with those waste streams EPA proposes for listing as RCRA hazardous wastes.

EPA's decision to exempt under the Bevill exclusion the "solid wastes" from DuPont's chloride-ilmenite plants was carried through in the July 1990 Report to Congress and the June 13, 1991 final rule (56 FR 27300) incorporating comments on the 1990 Report to Congress.

DuPont continues to support EPA's conclusion that these "coke and ore solids" are Bevill exempt and thus not subject to RCRA.

The Agency allows a mixture of a Bevill-exempt waste and a characteristically hazardous non-Bevill waste to maintain Bevill status so long as the resulting mixture does not exhibit any new characteristics that were not already exhibited by the Bevill portion. DuPont contends that the vanadium stream does not significantly increase concentrations of constituents over those that are already present in the Bevill portion alone. The Agency should consider revising the Bevill mixture rule at 40 CFR 261.3(a)(2) to address such scenarios when a mixture of a Bevill-exempt waste and a listed hazardous waste does not introduce any new constituents or increase constituent concentrations above that already present in the Bevill portion alone.

<u>Response 22-1d:</u> EPA acknowledges DuPont’s support. As discussed further in response to comment 22-3a-2, we are not promulgating the portion of the proposal that extended the scope of the listing to include solids derived from the vanadium stream. We are not listing any portion of the combined solids consisting of solids from the vanadium stream and “coke and ore” solids, which mingle before they leave the process. Since there is no mixture of separate waste streams, there is no need to modify or interpret the Bevill mixture rule. In addition, the Agency did not

propose to and is not promulgating any amendments to the Bevill mixture rule as part of today's rule. Any mixture of a Bevill exempt waste with a listed hazardous waste renders the mixture hazardous. The Agency restated the Bevill mixture rule at 63 FR 28595 (May 26, 1998). Today's rule does not alter in any way how the Bevill mixture rule applies to mixtures destined for disposal.

Comment 22-1e: DuPont concurs with EPA's position that solids in wastewaters from the reaction scrubbers are covered by the Bevill exemption

At 65 FR 55751 proposal, the Agency interprets the Bevill exemption to apply to solids from the "gases from the chlorinator" when such solids drop out of the scrubber waters to form sludges. DuPont supports this interpretation. These materials are waste solids from the production of titanium tetrachloride in the reaction process. DuPont agrees with the Bevill classifications for wastewater solids shown in the Titanium Dioxide Listing Background Document at pages 54 through 56. Examples of streams that fall into this category include solids from pretreated reaction area scrubber (RIN 1) and fume disposal/HCl scrubber (RIN 3) at the Delaware facility, pretreated reaction area scrubber water (RIN 2) at the Tennessee facility, and pretreated reaction scrubber water (RIN 3) at the Mississippi facility.

Response 22-1e: EPA acknowledges DuPont's support. We are clarifying, however, that only solids separated from scrubber waters originating in portions of the plant, namely titanium tetrachloride production, deemed to meet the definition of mineral processing (found in the September 1, 1989 Bevill rule) are Bevill exempt solid wastes. Based on our understanding of the process flow diagrams provided by the commenter, the four specific wastewaters identified in the comment are solely associated with the production of titanium tetrachloride; solids derived from these wastewaters would therefore be exempt. Once chemical manufacturing begins with the production of titanium dioxide, all scrubber liquids and solids subsequently generated are not subject to the Bevill exclusion and are fully subject to RCRA subtitle C if they are characteristically hazardous or listed.

Comment 22-1f: DuPont concurs with EPA that Off-Specification Titanium Dioxide Product does not warrant listing

At 65 FR 55765, the Agency proposes not to list off-specification titanium dioxide as a hazardous waste due to the very low risk posed by this material. DuPont concurs with this proposal. However, DuPont has commented elsewhere in Section 4 of these comments (see comment 22-5b) that the Agency's listing descriptions in the preamble discussion are not consistent with the proposed regulatory language.

Response 22-1f: EPA acknowledges DuPont's support. We are promulgating regulatory text that is more narrowly focused on solids associated with the production of ferric chloride. These revisions resolve the commenter's concern that the scope of the listing might inadvertently capture off-specification titanium dioxide.

Comment 22-1g: DuPont concurs with EPA's position that commingled wastewaters should not be listed

At 65 FR 55765, the Agency proposes not to list as a hazardous waste the commingled wastewaters from the chloride-ilmenite process due to the very low risk posed by this material. DuPont concurs with this proposal.

Response 22-1g: EPA acknowledges DuPont's support.

Comment 22-1h: DuPont concurs and supports the Agency's determination and rationale for not listing waste from inorganic HCN production

DuPont agrees with and supports the Agency's decision not to list inorganic hydrogen cyanide (HCN) production wastes in this proposed rulemaking and further supports the Agency's decision not to include in the category hydrogen cyanide wastes produced by organic production processes. DuPont believes that the information collection request [3007] Survey responses and the sampling conducted at our Memphis, Tennessee facility support the EPA's conclusion that a new regulation dealing with a separate listing of individual waste streams does not meet the criteria set forth in 40 CFR 261.11(a)(3) for listing wastes as hazardous. The wastes from these facilities are effectively managed in accordance with long established requirements under RCRA and the Clean Water Act in on-site wastewater treatment processes, industrial landfills, municipal landfills, hazardous waste incinerators, hazardous waste landfills, and hazardous waste injection wells based on their exhibiting or not exhibiting reactivity or other characteristics. Other residuals are produced in the facilities and recycled back to the production process via enclosed piping systems and tanks, minimizing the potential for environmental releases. Therefore, DuPont concurs with EPA's assessment that handling of solid wastes and residuals in accordance with current regulatory requirements from DuPont's HCN manufacturing facilities does not pose any present or potential hazard to human health or the environment.

Response 22-1h: EPA acknowledges DuPont's support.

Comment 22-2: EPA Mistakenly Differentiates Between the Chloride Process and Chloride-ilmenite Process after Considering These Two Processes Part of the Chloride Process in Previous RCRA Bevill Rulemakings

EPA has proposed to list as hazardous waste K178 numerous nonwastewater solids from DuPont's chloride-ilmenite process. In proposing to list these nonwastewater solids, EPA suggested that they are not Bevill-exempt. In contrast, EPA has proposed not to list any nonwastewater solids from the chloride process, even though the nonwastewater solids generated derive from the same process, have essentially the same chemical composition, and are subject to the same management. EPA also suggested that the solids from the chloride process are Bevill-exempt, even though they are generated by the same processing steps employed in the chloride-ilmenite process. This differential treatment is unjustified, and conflicts with EPA's prior Bevill determinations in which EPA provided similar treatment for the chloride and chloride-ilmenite processes. In fact, before this listing proposal, EPA evaluated these processes as a single category despite DuPont's urging to view them differently, and recognized that the waste solids streams from these processes were "functionally identical." 55 FR 2329/3 (Jan. 23, 1990). The proposal thus unfairly provides different and less favorable treatment to wastes generated in DuPont's chloride-ilmenite process. To be fair, the final listing rule should treat DuPont's waste similarly to those from the chloride process, and thus should not list any waste streams from the chloride-ilmenite process.

Comment 22-2a: Technical Similarity of the Chloride and Chloride-Ilmenite Processes and Wastes

The chloride process and the chloride-ilmenite process are two processes within the same industry that are employed to make the same products - intermediate product titanium tetrachloride and final product titanium dioxide. The chloride process uses as a feedstock a higher-grade "rutile" or "ilmenite" ore, and consequently produces waste streams that are marginally smaller in volume and that are marginally less concentrated. Except for iron, which is present in higher concentrations in lower grade ilmenite ore, the wastes generated by the two processes are virtually identical, and the processes also are essentially identical. The processes employ the same unit operations, and produce similar waste solids streams having similar waste characteristics that are managed in similar ways.

As the proposal recognizes, the chloride process generates: (1) process and non-process wastewaters; (2) Bevill-exempt solids from titanium tetrachloride production; (3) vanadium waste generated in the purification process; (4) waste sands from finishing and oxidation units; and (5) wastewater treatment sludges (which may be derived from commingled Bevill-exempt and non-exempt streams). 65 FR 55748/3-55749/1. The proposal also identifies but does not discuss (6) "waste acid, which they mingle with coke and ore solids before treatment." 65 FR 55748/2. The proposal identifies similar "wastes" for the chloride-ilmenite process, but adds numerous wastes not identified for the similar chloride process and suggests a different Bevill-exempt status for these other chloride-ilmenite streams: (1) process and non-process wastewaters; (2) Bevill-exempt solids from titanium tetrachloride production; (3) vanadium waste generated in the purification process; (4) waste sands removed from the reactor purge stream; (5) nonexempt nonwastewaters, including wastewater treatment solids and ferric chloride filtration solids; (6) "waste acid" ferric chloride; (7) spent scrubber waters from reaction fume disposal; (8) HCl from the reaction scrubber; (9) additive feeder vent filter solids; (10) off-specification titanium dioxide product; and (11) rail car product washout wastewater. 65 FR 55749/2-3.

The greater number of wastes identified for the chloride-ilmenite process reflects EPA's substantial knowledge gleaned from the history of Bevill determinations, which in turn led

EPA to request and obtain much more detailed responses from DuPont for the chloride-ilmenite process than for the chloride process.[1] In fact, however, these processes generate similar waste streams. These similar waste streams are pulled from the 3007 submissions from each producer and the "Titanium Dioxide Listing Background Document For Inorganic Chemical Listing Determination" page 10 and 11. Table 2.1 is summary of key process streams that contain the listed metals.

Table 2.1
Waste Reported by Titanium Dioxide Facilities

	Commingled Wastewater Sludge	Off-gas Scrubbing solids	"Bevill Exempt Waste"
Chloride Ilmenite			
Delaware	To Iron Rich^{TM}	RIN 2	Iron Rich^{TM}
Mississippi	RIN 108	RIN 3	RIN 16
Tennessee	RIN 108	RIN 2	RIN 9
Chloride			
Georgia	RIN 14	RIN 7	RIN 6
Louisiana	RIN 5	RIN 2	RIN 4 (to RIN 6)
Maryland	RIN 4	RIN 2	RIN 1 (to RIN 4)
Ohio	RIN 4	RIN 2	RIN 1 (to RIN 4)

Table 2.2 below is a comparison of Tennessee Plant RIN 108 and RIN 14 for the Georgia Plant. Table 2.5 is a comparison of Iron Rich^{TM} from the Delaware Plant and RIN 4 from the Maryland Plant.

---

[1] See Titanium Dioxide Listing Background Document for the Inorganic Chemical Listing Determination, August, 2000; 1998 RCRA § 3007 Survey of the Inorganic Chemicals Industry - E.I. DuPont de Nemours, DeLisle - Pass Christian, MS response; 1998 RCRA § 3007 Survey of the Inorganic Chemicals Industry - E.I. DuPont de Nemours & Company - Edge Moor, DE response, May 14, 1999; 1998 RCRA § 3007 Survey of the Inorganic Chemicals Industry - E.I. DuPont de Nemours & Co., New Johnsonville, TN response, May 27, 1999 in Docket Number F-2000-ICMP-FFFFF, Docket Item Numbers 330, 333, 334, and 335, respectively. See also 1998 RCRA § 3007 Survey of the Inorganic Chemicals Industry Kemira Pigments, Inc.- Savannah, Georgia response (redacted); 1998 RCRA § 3007 Survey of the Inorganic Chemicals Industry -Millennium Inorganic Chemicals, Inc. Baltimore, Maryland response (redacted); 1998 RCRA § 3007 Survey of the Inorganic Chemicals Industry - Kerr McGee Chemical Corp.- Hamilton, Mississippi response; 1998 RCRA § 3007 Survey of the Inorganic Chemicals Industry - Kronos/Louisiana Pigment Co. - West Lake, Louisiana (redacted); 1998 RCRA § 3007 Survey of the Inorganic Chemicals Industry - Millennium Inorganic Chemicals, Plant 1 - Ashtabula, OH response (redacted); and 1998 RCRA § 3007 Survey of the Inorganic Chemicals Industry - Millennium Inorganic Chemicals, Plant 2 - Ashtabula, OH response (redacted); which are Docket Item Numbers 331, 332, 253, 338, 339 and 340, respectively.

Table 2.2
EPA 3007 Sampling - Inorganic Chemicals - Nonwastewaters

	KP-SO-02			DPN-SO-01		
	Total, mg/kg	TCLP, mg/L	SPLP, mg/L	Total, mg/kg	TCLP, mg/L	SPLP, mg/L
Aluminum	4520	<1	<0.1	5770	<1	<0.1
Antimony	0.8	<0.5	0.013	0.7	<0.5	0.021
Arsenic	2.4	<0.5	<0.0035	2.8	<0.5	<0.0035
Barium	40.2	<2	0.078	49.6	<2	0.12
Beryllium	0.4	<0.02	<0.002	0.5	<0.02	<0.002
Boron	16.9	3.0	0.28	24.5	<2	0.45
Cadmium	<0.5	<0.05	<0.005	<0.5	<0.05	<0.005
Calcium	15600	551	50.8	1500	44.2	14.0
Chromium	712	<0.05	0.001	499	0.06	<0.005
Chromium +6	<0.40	NA	<0.02	<0.4	NA	<0.02
Cobalt	2.4	<0.05	<0.005	7.0	0.13	<0.005
Copper	12.6	<0.25	0.004	15.8	<0.25	0.003
Iron	36500	<1	<0.05	63200	567	2.2
Lead	42.4'	0.05	0.001	42.4	<0.5	0.002
Magnesium	5110	197	38	769	21.9	8.0
Manganese	3130	7.8	<0.005	2890	47.4	1.5
Mercury	<0.1	<0.002	<0.0002	0.2	<0.002	<0.0002
Molybdenum	10.6	<0.2	0.093	4.5	<0.2	0.006
Nickel	47.1	0.13	<0.005	59.8	1.2	0.007
Potassium	109	<10	3.4	<100	<10	4
Selenium	<0.5	<0.5	<0.005	0.5	<0.5	<0.005
Silver	1.5	<0.1	<0.001	0.2	<0.1	<0.001
Sodium	1540	NA	85.4	4920	NA	267
Thallium	8.1	<2	<0.0022	7.2	<2	<0.00225
Tin	75.2	<0.5	<0.01	12.9	<0.5	<0.01
Titanium	8310	<0.05	<0.005	5360	<0.05	<0.005
Vanadium	1570	0.03	0.039	1060	<0.05	<0.005
Zinc	31.5	0.4	0.02	57.2	0.53	0.073
Process Material	WWT Sludge (RIN 14)			Wastewater Treatment Sludge		
Process Type	Commingled Chloride & Sulfate			New.Johnsonville		
Management Practices	Dredged from impoundment, filter pressed, drainage piles, placed in on-site industrial landfill			Dewatered and placed in on-site landfill		

	KP-SO-02			DPN-SO-01		
	Total, mg/kg	TCLP, mg/L	SPLP, mg/L	Total, mg/kg	TCLP, mg/L	SPLP, mg/L
Site/Company	Kemira			New Johnsonville		
Reference	Ref. 1, Table 3.19; Ref. 2 Lab Report			Ref. 1, Table 3.27, Ref. 3, Lab Report		
*Denotes a discrepancy between the Background Document and the Sampling and Analytical Data Report						
1- Titanium Dioxide Listing Background Document for the Inorganic Chemicals Listing Determination, August, 2000, Tables 3.19 and 3.27, Docket F-2000-ICMP-FFFFF, Docket Item Number 330						
2 - Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes from the Inorganic Titanium Dioxide Manufacturing Sector, Kemira Pigments, Inc., Savannah, Georgia, September 9, 1999, Appendix C, Analytical Data, Docket F-2000-ICMP-FFFFF, Docket Item Number 353						
3 - Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes from the Inorganic Titanium Dioxide Manufacturing Sector, E.I. du Pont de Nemours and Company, New Johnsonville, Tennessee, September 14, 1999, Appendix C, Analytical Data, Docket F-2000-ICMPFFFFF, Docket Item Number 354						

Of greater importance, as discussed below, the proposal provided different and less favorable treatment to the similar streams that it identified, without any justification for doing so in the record.

The waste streams generated by both processes have essentially identical chemical compositions, except for the concentration of iron chloride. For example, as EPA recognized, waste acids are generated in both chloride and chloride-ilmenite processes. These waste acids have essentially the same metal composition, as demonstrated by the data provided below in Table 2.3 and Table 2.4 which is from typical data generated at DuPont chloride and chloride ilmenite processes (note the data is presented as oxide feed to the titanium tetrachloride process). The only measurable difference (within the variability of different ilmenite, rutile and slag ores) is the iron levels. In each site this iron chloride is separated and managed differently as are the Bevill exempt coke and ore solids.

Separation of the iron chloride "waste acid" from the coke and ore solids is accomplished in a similar manner at the Tennessee and Mississippi Plants. The Mississippi Plant separates out coke and ore solids and eventually underground injects the solution. The Tennessee Plant separates coke and ore solids from the "waste acid" and sends the "waste acid" to the sodium chloride process. At both the Mississippi and Tennessee Plant the coke and ore solids are "dewatered" and placed in a landfill.

The Delaware Plant, separates solids in several stages, in the concentrated iron stage chlorine is injected to convert residual ferrous chloride to ferric chloride producing a commercial product. In the initial stage the majority of the coke and ore solids are slurried with water and later filtered to create Iron Rich^{TM}. Delaware coke and ore solids, which are represented by Iron Rich^{TM}, were the only "Bevill" - exempt solids sampled by the Agency. Section 5.2, Table 5.2 of these comments contains data collected by DuPont on the contribution of Mn and Tl from the wastewater treatment sludge to Iron Rich^{TM}, which is negligible.

What the Agency has done in sampling RIN 4 at the Maryland Facility and Iron Rich^{TM}, is compare the Bevill exempt solids and waste acid streams that are neutralized at these two facilities. The Iron Rich^{TM} has the same characteristics as the chloride process filter cake from the Maryland facility as shown in Table 2.5.

In each chloride-ilmenite case, once the iron chloride is separated from the Bevill solids, the residual "waste acid" that is attached to the Bevill exempt solids is analogous to the chloride process "waste acid" commingled with Bevill exempt solids. As shown in Table 2.3 and Table 2.4 once the ore is chlorinated in the chloride-ilmenite process the resulting waste acid has similar characteristics to the waste acids which separate out in the chloride process, prior to "processing" as a filter cake from mechanical separation devices, or a gravity separation sludge.

Table 2.3

Typical Chloride Process
Pounds of metal oxide/100 pounds Ore

Component	Ore Feed to Reactor	Separated Acid, Measured as Oxide Equivalents to Metal Chlorides
Ti02	95	2
Fe203	≤1	≤1
Mn0	≤2	≤2
Cr203	≤0.5	≤0.5
V205	0.4	0.4
Mg0	≤1	≤1

Table 2.4

Typical Chloride Ilmenite Process
Pounds of metal oxide/ 100 pounds Ore

Component	Ore Feed To Reactor A	Separated Acid, Measured as Oxide Equivalents to Metal Chlorides
Ti02	67	3
Fe203	28	28
Mn0	≤1	≤1
Cr203	≤0.2	≤0.2

V205	0.15	0.15
Mg0	≤0.5	≤0.5

For another example, EPA's own sampling data demonstrated the similar composition of chloride-process wastewater treatment sludges to certain non-exempt nonwastewater solids from the chloride-ilmenite process. Samples of the chloride process sludges were obtained at Millennium's Baltimore Maryland facility. Samples of the non-exempt nonwastewater solids were obtained from the Iron Rich$^{\text{TM}}$ at DuPont's Edge Moor, Delaware facility. The Iron Rich$^{\text{TM}}$ is composed of Bevill-exempt coke and ore solids and wastewater treatment sludges. The results are very similar in regard to concentrations of all of the constituents of toxicological concern for listing at the leachable levels identified as seen in Table 2.5.

Table 2.5
EPA Sampling Data - Inorganic Chemicals

	MI-SO-01			**DPE-SO-01**		
	Total, mg/kg	TCLP, mg/L	SPLP, mg/L	Total, mg/kg	TCLP, mg/L	SPLP, mg/L
Aluminum	8740	<1	0.24	10100	<1	<0.1
Antimony	0.6	0.6	0.006	0.9	<0.021	0.02
Arsenic	1.6	<0.5	0.00005	2.2	<0.035	0.001
Barium	49.8	<2	0.06	178	2.4	0.92
Beryllium	1 .5	<0.02	<0.002	1.2	<0.00024	<0.002
Boron	9.4	<2	0.15	30.0	1.7	0.61
Cadmium	<0.5	<0.05	<0.005	0.6	<0.0013	<0.005
Calcium	7490	403	294	28500	1330	1230
Chromium	1230	<0.05	0.001	777	<0.05	0.002
Chromium +6	<0.40	NA	<0.02	<0.40	NA	<0.02
Cobalt	2.6	0.056	<0.005	44.5	0.43	<0.005
Copper	16.7	<0.25	<0.002	28.5	0.014	0.003
Iron	62700	553	<0.05	91600	348	0.18
Lead	1.3	0.5	0.002	309	0.03	0.003
Magnesium	14400	569	30.4	3140	61.3	33.4
Manganese	12700	468	2.63	10600	252	16.3
Mercury	<0.1	<0.002	3.5E-05	<0.1	<0.002	<0.0002
Molybdenum	1.6	<0.2	0.013	7.4	0.026	0.005
Nickel	59.9	0.5	<0.005	91.8	0.5	<0.005
Potassium	<100	<1	1.1	140	<10	1.8

Table 2.5
EPA Sampling Data - Inorganic Chemicals

	MI-SO-01			DPE-SO-01		
	Total, mg/kg	TCLP, mg/L	SPLP, mg/L	Total, mg/kg	TCLP, mg/L	SPLP, mg/L
Selenium	<0.5	<0.5	<0.005	<0.5	<0.5	<0.005
Silver	0.6	<0.1	<0.001	<0.1	<0.1	<0.001
Sodium	671	NA	29	770	NA	41.8
Thallium	3.0	<2	0.003	3.7	0.28	0.012
Tin	2.7	<0.5	<0.01	53.2	0.025	<0.01
Titanium	5270	<0.05	<0.005	6380	<0.05	<0.005
Vanadium	2320	<0.05	0.004	240	0.0003	<0.005
Zinc	<5	<2	0.025	122	1.1	0.03
Process Material	WWT Sludge			Iron Rich		
Process Type	Commingled Chloride & Sulfate			Chloride-Ilmenite		
Management Practices	Dredged from impoundment, filter pressed, placed in on-site industrial landfill			Stored in piles, marketed as Iron Rich		
Site/Company	Millennium, Baltimore			DuPont, Delaware		
Reference	Ref. 1, Table 3.19; Ref. 2 Lab Report			Ref. 1, Table 3.27; Ref. 3, Lab Report		

References:

1 - Titanium Dioxide Listing Background Document for the Inorganic Chemicals Listing Determination, August, 2000, Tables 3.19 and 3.27, Docket F-2000-ICMP-FFFFF, Docket Item Number 330

2 - Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes from the Inorganic Titanium Dioxide Manufacturing Sector, Millennium Inorganic Chemicals, Inc., Baltimore, MD, September 23 and 30, 1999, Appendix C, Analytical Data, Docket F-2000-ICM P-FFFFF, Docket Item Number 355

3- Sampling and Analytical Data Report for Record Sampling and Characterization of Wastes from the Inorganic Titanium Dioxide Manufacturing Sector, E.I. du Pont de Nemours and Company, Edge Moor, Delaware, September 7, 1999, Appendix C, Analytical Data, Docket F-2000-ICMP-FFFFF, Docket Item Number 356

Finally, the chloride and chloride-ilmenite process streams are managed in essentially similar ways. Most of the wastes generated are managed by further processing into products, on-site treatment, and on-site land disposal or deepwell injection. However, some wastes from some facilities using the chloride process land dispose of wastes offsite (despite EPA's belief that such wastes from chloride process plants are only managed on-site - 65 FR 55753/1), and DuPont sells Iron Rich for off-site land application.

DuPont's analysis of our competitor's waste disposal practices, from public documents suggests disposal practices different than stated by the Agency in the proposed listing in Table 111-39, 65 FR 55749:

<u>Waste Category</u> <u>Management Practices</u>

1. Chloride process solids (Bevill exempt) 65 FR 55753/1	Though stated as disposed of in on-site impoundments and on-site Industrial Subtitle D landfills at least one producer disposes of in an off-site industrial landfill.
2. Waste sands from oxidation, milling and scouring. 65 FR 55753/3	Though stated as handled in both on- and off-site industrial Subtitle D landfills, at least one producer has sent (1999) this waste stream to a municipal landfill.
3. Gypsum from sulfate process 65 FR 55754/2	Though stated as disposed of in on-site Industrial Subtitle D landfill at least one producer disposes of in an off-site unlined landfill which is contiguous with a closed hazardous waste landfill.
4. Wastewater treatment sludges from commingled chloride and sulfate process(partially Bevill exempt). 65 FR 55758/2	Though stated as disposed of in on-site Industrial Subtitle D landfill at least one producer has disposed (and currently is?) in an off-site landfill.

Documentation of these assertions can be found in Attachment 2.1 with the circled numbers referring to the 4 streams above.

Since EPA modeled these wastes using on-site disposal as the criteria for model inputs it has compromised the results of these analyses by the incorrect nature of the model inputs. We believe EPA should remodel these wastes using actual disposal methods to determine whether their conclusions are correct. EPA should use the same approach to modeling factors for these off-site practices as they ultimately do for the chloride-ilmenite process off-site practices.

<u>Response 22-2a:</u> We acknowledge the commenter's argument that the Bevill-exempt solids from the chloride and chloride-ilmenite process are likely to be quite similar, as has been previously concluded by the Agency in our mineral processing determinations. As finalized, however, the listing only impacts solids that are associated with the production of ferric chloride, and thus the listing has no impact on facilities using the chloride process (and in fact only potentially impacts the DuPont Edge Moor facility). We are not promulgating the portion of the proposed listing that would have captured solids associated with the vanadium stream because we now have more complete information regarding the nature of this stream and understand that this stream exits the condenser unit along with the coke and ore solids. We are also not promulgating the portion of the proposed listing for the wastewater treatment solids. As a result, the commenter's concern about the unfair application of the listing solely to the chloride-ilmenite process, while never valid, is now moot as well.

We assessed the titanium dioxide industry carefully, and attempted to fairly apply the listing criteria to all wastes generated in order to fully comply with the requirements of the EDF consent decree. In assessing this industry, we looked for logical ways to combine facilities and waste generation practices to avoid inappropriate aggregations that might mask important waste differences. We found clearly that wastes generated from the sulfate process differed from the wastes generated by the chloride and chloride-ilmenite processes. These processes differ fundamentally in their process chemistry and therefore generated discrete and different wastes. While the differences between the chloride and chloride-ilmenite processes are more subtle (as described above by the commenter), these differences were sufficient basis for us to assess the processes independently to determine if they generate wastes that warrant listing.

The most important difference between the chloride and chloride-ilmenite processes is the elevated iron levels in the chloride-ilmenite feedstock. After chlorination, subsequent separation from the titanium tetrachloride process stream, and quenching, the resultant iron chlorides form a highly concentrated ferric chloride acid stream in the chloride-ilmenite process. While the chloride process also generates ferric chlorides, their concentration is significantly lower. Chloride facilities do not generate a segregated ferric chloride waste acid stream; chloride facilities generally commingle their scrubber waters and quench waters (containing the ferric chlorides) and subject the mixture to wastewater treatment (including neutralization). The chloride-ilmenite plants, in contrast, each isolate their concentrated acid stream and manage this stream as a discrete waste (one facility disposes of their acid as hazardous waste via deep well injection, the second facility produces sodium chloride from its acid stream, and the third process the acid and sells it as a treatment agent for water and wastewater treatment). We structured our listing determination to ensure that we could adequately assess these unique wastes.

We note also that we conducted extensive field work and risk assessment screening and modeling at the titanium dioxide plants that use the chloride and sulfate processes, in fact collecting twice as many samples from the chloride and sulfate plants as from the chloride ilmenite plants, as summarized in Table 22-1:

Table 22-1. Summary of EPA Sampling in the Titanium Dioxide Sector

Titanium Dioxide Sector	Waste Category	EPA Sample ID Number
Sulfate process	Digestion sludge	MI-SO-02 KP-SO-03
	Gypsum	MI-SO-04 MI-SO-03 KP-SO-01
	Digestion scrubber wastewater	MI-WW-03
Chloride process	Scouring sand	KP-SO-04
	Wastewaters	KM-SI-01
	Commingled solids and acid	MI-WW-01

Table 22-1. Summary of EPA Sampling in the Titanium Dioxide Sector

Titanium Dioxide Sector	Waste Category	EPA Sample ID Number
	Scrubber water	MI-WW-02
	Impoundment solids	KM-SI-04
Chloride and sulfate process	Milling sand	KP-SO-05
	Mixed wastewater treatment solids and Bevill exempt solids	MI-SO-01 KP-SO-02
	Mixed wastewaters	KP-WW-01 MI-WW-04
Chloride-ilmenite process	Waste acid	DPE-WW-03 DPN-SO-03
	Off-spec titanium dioxide	DPN-SO-02
	Commingled wastewaters	DPE-WW-01 DPN-WW-01
	Commingled wastewater treatment solids and Bevill exempt solids	DPE-SO-01 DPN-SO-01

Our approach to assessing the chloride and chloride-ilmenite process wastes was the same, although the listing determination resulting from these assessments differed for the two sectors as a result of the differing risks we identified. Our approach, applicable to both processes, was first to determine which wastes were Bevill exempt and therefore outside the scope of the listing determination. Almost all of the TiO2 manufacturers generated a solid stream that was comprised of exempt and non-exempt components. We then attempted to focus our risk assessment activities on the wastes with the most significant non-exempt components. We used the assessments at these facilities as surrogates for the facilities with less significant non-exempt components. For example, our data indicated that the two Millennium plants in Ashtabula, Ohio generated solids that appeared to be almost entirely exempt. We instead assessed the Millennium Baltimore and Kemira Savannah plants' wastes which contained much higher proportions of non-exempt solids. If we had found risk supporting a listing at either of these plants, we would have proposed to create a listing for the non-exempt portion of all the chloride process plant solids, including the Millennium Ashtabula plants' solids. This extension would have been comparable to our proposal to list all non-exempt chloride-ilmenite process solids, not just the modeled solids at Edge Moor.

To summarize, we proposed different results for the chloride and chloride-ilmenite process wastes primarily because we identified different plausible management scenarios for these wastes and the risks associated with these scenarios supported different listing determinations. In addition, we note that there are some differences in the waste compositions that indicate on an inherent hazard basis (i.e., not accounting for plausible management) that the chloride-ilmenite waste contain somewhat higher levels of the constituents that were the focus of our risk analysis. The following table summarizes our analytical results for these constituents:

EPA Sampling Data Comparing SPLP Results (mg/L) for the Millennium Baltimore and DuPont Edge Moor Wastes		
	Millennium Baltimore (MI-SO-01)	DuPont Edge Moor (DPE-SO-01)
Antimony	0.006	0.02
Arsenic	0.00005	0.001
Manganese	2.63	16.3
Thallium	0.003	0.012

This table shows that the DuPont solids leached more readily than the Millennium solids for each of the constituents of concern when tested with the SPLP. The SPLP is the leaching procedure we used as an appropriate measure of mobility in an industrial solid waste landfill scenario. Both the chloride and chloride-ilmenite wastes were assessed via this scenario (with the additional difference of on-site assumptions for the chloride industry wastes and off-site assumptions for the chloride-ilmenite wastes). We note that manganese was a constituent of concern in our proposal only. We are deferring action on manganese for the final rule. See Section IV.B. of the preamble. The table shows that, had manganese remained a constituent of concern, we would have concluded that the DuPont solids leached manganese more readily than the Millennium solids.

The remainder of this response addresses the commenter's assertion that EPA should have modeled off-site, as well as on-site waste management scenarios for various chloride and sulfate process wastes. The commenter argued that it was unfair to only assess the proposed K178 wastes for potential off-site management. Each of the four potential "off-site scenarios" are discussed below.

Chloride process solids, Millennium Ashtabula, OH facilities

DuPont provided information from public sources that indicates that one chloride process facility (Millennium Ashtabula, OH facility) has disposed of chloride process solids in off-site industrial landfills, in contrast with the on-site modeling assumed by and modeled by EPA.

As described in the proposal, we assessed this facility (actually 2 adjacent plants owned by Millennium) and determined that their waste was likely to contain very little non-exempt material (65 FR 55753). We used our modeling of the commingled chloride and sulfate process solids as a surrogate for these plants' solids, all of which were reported to be landfilled on site. In addition, we assessed the on-site management of wastewaters at these plants (see p. 16 of the Titanium Dioxide Listing Background Document, August 2000) and determined that there were no drinking water wells likely to be impacted by the facilities' reported on-site waste management activities in this heavily industrialized location where the groundwater flow direction is away from known populated areas.

In apparent contrast with the information upon which we based our proposal, the commenter provided information indicating that Millennium disposed of their chloride solids in two *off-site* landfills. The information was documented in a memorandum from Elizabeth Wilson of Ohio EPA dated November 8, 2000 (see Attachment 2.1 of the comments). The memorandum documents the possible historical disposal of the chloride solids in two different off-site landfills: the Waste Management Geneva (formerly named Dougherty) Landfill prior to 1991 and the Reserve Environmental Services (RES) landfill between 1991 and 1995. The solids (from both plants) have been disposed of in the Millennium landfill from 1995 to the present.

At the time of the proposal, we were not aware of the historical off-site disposal of the chloride solids from the Millennium Ashtabula plants. We identified the disposal in the current on-site Millennium landfill based on responses to the §3007 survey, in which the base reporting year was 1998.

Since the Millennium on-site landfill is a relatively new landfill (1995) with plenty of remaining capacity, we believe it is entirely reasonable to assume that Millennium will continue to use its on-site landfill to manage the waste solids from the two adjacent Millennium plants. Millennium used the RES facility, an industrial landfill, as an interim management practice while their on-site landfill was being developed.[2] The Geneva landfill, located approximately 11 miles west of the plant, may have been used prior to construction of the newer and nearer RES landfill, although we were unable to confirm this. Even if we had known about these historical management practices at this site, we would have likely limited our assessment to the captive Millennium landfill, and assumed that Millennium would use it's own landfill capacity (and capital investment) rather than incur the costs of off-site commercial landfilling.

Scouring Sand, Kerr McGee Savannah, Georgia
DuPont provided information from public sources that, although EPA assumed a waste management practice of both on- and off-site industrial Subtitle D landfills, at least one producer (Kerr-McGee, formerly Kemira, Savannah, GA facility) disposes of waste scouring sand in a municipal landfill.

For the proposal, we assessed a dedicated on-site landfill for scouring sand, reflecting the type of management reported for the waste. We assessed the groundwater ingestion pathway for the on-site landfill scenario, which screened out when we compared the SPLP results for this waste directly to the health-based levels. We also assessed an off-site industrial landfill scenario and found that this scenario similarly posed no risk.

The commenter provided information indicating that Kerr-McGee disposed of their scouring sand in two off-site landfills (Superior Sanitation Services Landfill and Savannah Regional Industrial Landfill). The information was documented in a table titled “Attachment 2. Kemira Solid Waste Disposal - Feb thru May 1999.” See Attachment 2.1 of DuPont’s comments. The table shows the

---

[2] See Memo to File from Max Diaz, Chemical Engineer, Office of Solid Waste, re Millennium Inorganic Chemicals (MIC) Landfill; Ashtabula, OH.

historical disposal of the scouring sand in the two different off-site landfills between March 18, 1999 through May 25, 1999.

Based on recent information from Kerr-McGee Pigments (formerly Kemira), we have verified that scouring sand is disposed of in an off-site industrial Subtitle D landfill that is also permitted to accept municipal waste[3]. In response to this new information, we assessed whether our TCLP results for this waste indicate that the municipal landfill scenario would pose risk. This assessment, summarized below, showed that the TCLP concentrations for the toxic constituents detected in the scouring sand are generally lower than their respective health-based levels (HBLs) and thus screen out. Two constituents, boron and lead, exceed their HBLs by a factor of 2 or less, and using the same assumptions used in the proposal regarding low level contamination (see 65 FR 55696), we assume they screen out. See Table 22-2 below:

Table 22-2: Initial Screening Analysis for Scouring Sand

Constituent	**KP-SO-04 SPLP (mg/L)**	**KP-SO-04 TCLP (mg/L)**	**HBL (mg/L)**	**AWQC (mg/L)**
Aluminum	0.23	1.2	16	0.087
Antimony	0.007	<0.5	0.0063	0.014
Barium	0.11	<2	1.1	1
Boron	0.15	1.7	1.4	NA
Calcium	0.96	2.3	NA	NA
Chromium	0.018	0.03	23	0.74
Copper	0.004	<0.25	1.3	0.0090
Iron	<0.05	<1	5	1
Lead	0.001*	0.03	0.015	0.0025
Magnesium	0.066	0.8	NA	NA
Manganese	0.006	<0.05	0.73	0.05
Mercury	0.0004	<0.002	0.0047	0.000050
Nickel	0.019	<0.2	0.31	0.052
Sodium	8.4	NA	NA	NA
Tin	<0.01	<0.5	9.4	NA
Titanium	0.068	<0.05	NA	Na
Zinc	0.067	0.7	4.7	0.12

---

[3] Memo to file from Maximo Diaz, EPA, “Kerr-McGee Pigments, Savannah, GA”, dated June 28, 2001.

*Results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits.
< - Analyte not detected above the laboratory's established reporting limit or method.

Therefore, although we did not evaluate the municipal landfill scenario for the scouring sand in the proposal, our screening analysis indicates the new scenario identified by the commenter does not present risk. We note that, although manganese was a constituent of concern in our proposal, we are deferring action on manganese for the final rule. See Section IV.B. of the preamble. We also note that, if manganese had remained a constituent of concern, and if we had used the same RfD and HBL that we used in the proposal, we would have concluded that manganese in these materials did not pose significant risks.

Gypsum, Millennium Baltimore, Maryland

DuPont provided information from public sources that, although EPA assumed a waste management practice of on-site industrial Subtitle D landfilling, at least one producer (Millennium Baltimore, MD facility) disposes of secondary gypsum from the sulfate process in an off-site unlined landfill which is contiguous with a closed hazardous waste landfill.

Based on recent information obtained from Millennium officials,[4] we have confirmed that the gypsum is, in fact, disposed of in a different landfill other than the one reported by the generating facility (HPP Landfill) and on which the risk modeling in the proposal is based.

The newly identified unit, the Quarantine Road Landfill, is also owned by Millennium and is located 3,000 feet northwest of their on-site HPP Landfill. Based on the volumes reported by Millennium to be placed in this landfill in 1999,[5] it appears that virtually all of the secondary gypsum is being landfilled in this unit. The unit is an industrial waste landfill similar to the on-site unit that is used solely for the purpose of disposing of Millennium's secondary gypsum (captive landfill); the secondary gypsum is occasionally sold to a wallboard manufacturer during favorable market conditions.

It is unlikely that the secondary gypsum currently being disposed of in the Quarantine Road landfill will be disposed of in an off-site commercial landfill because (1) there is ample remaining capacity and (2) the potential wallboard-marketing opportunity. Therefore, we assessed the potential risk associated with the newly identified Quarantine Road landfill on a site-specific basis similar to the approach we used for the HPP landfill. We compared the distance between the nearest residence and the Quarantine Road landfill with the distance between the nearest residence and the HPP landfill, for which detailed risk modeling was performed in the proposal. Both landfills represent the same industrial landfill management scenario.

The distance between the Quarantine Road landfill and the nearest residence is approximately

---

[4] See memo to docket from Max Diaz, Chemical Engineer, Hazardous Waste Identification Division, EPA, dated August 7, 2001.

[5] See letter to Max Diaz from Frank Martin, Millennium Inorganic Chemicals, dated December 6, 2000.

2,500 feet to the southwest. This distance is the same as the distance between the HPP and the nearest resident (2,500 feet to the south). Since the distance from the new Quarantine Road landfill to the nearest residence is identical to what we modeled for the HPP landfill in the proposal, which resulted in a no-list decision, our proposed decision for this waste is intact. It should also be noted that since the area is zoned as heavy industrial, the residences are required to use the available city water supply.

This is a minor extension of the logic we used for the proposal. We believe a nearby captive landfill can be assessed in the same way as an on-site landfill because of the incentives for the facility to continue using that landfill. In this case, we have not identified any additional risk associated with this newly identified landfill.

Commingled chloride/sulfate solids, Kerr McGee, Savannah, Georgia

DuPont provided information from public sources that, although EPA assumed a waste management practice of on-site industrial Subtitle D landfilling, at least one producer [Kerr-McGee (formerly Kemira) Savannah, GA facility] disposes of commingled chloride/sulfate process wastewater treatment sludges in an off-site landfill.

In the proposal, the Kerr-McGee wastewater treatment sludge waste screened out when the reported on-site industrial landfill scenario was assessed by comparing the SPLP results of the detected constituents to their health-based risk levels.

The commenter provided information indicating Kerr McGee disposed of their WWT solids off-site at the Little Neck Road Landfill in Savannah, GA, owned and operated by Chemical Waste Management, Inc. The information was documented in an May 5, 1993 Kemira, Inc. Trip Report prepared by the Georgia Department of Natural Resources.

Recent information from Kerr-McGee (current owner of the Kemira plant) verifies that off-site disposal did occur on an interim basis during 1993, with no off-site disposal occurring since January 1, 1994. Kerr-McGee identified the landfill as the Superior Landfill and Recycling Center, 3001 Little Neck Road, Savannah, GA. The new operator is unclear as to why this off-site disposal occurred since their current on-site landfill was in operation during that time. The newly identified off-site landfill is an Subtitle D unit that is permitted to accept both industrial and municipal waste. The landfill does accept municipal waste and is co-disposing both industrial and municipal waste in the same cells. Since (1) Kerr-McGee has no plans to use future off-site disposal, (2) the off-site landfill disposal was only used on an interim basis, we have concluded that the on-site landfill disposal scenario assessed in the proposal is still valid. EPA may in the future revisit this decision if the waste is further managed in the Superior landfill or any other off-site industrial/municipal landfill.

After consideration of the new information provided by the commenter regarding the potential for off-site management of chloride and sulfate process wastes, we continue to believe that our proposal not to list these wastes was appropriate. The municipal landfill scenario identified by the commenter for Kerr McGee's scouring sand poses no additional risk. The off-site management at the nearby landfill for Millennium Baltimore's gypsum poses no additional risk beyond the management scenario we assessed for the proposal. In addition, we have no information to cause us to expect that the historical off-site management at Millennium Ashtabula and Kemira Savannah is likely to resume soon given that both facilities have on-site landfill capacity. If off-site management were to resume, we may re-evaluate our listing determination. In contrast, the DuPont Edge Moor plant has no on-site landfilling capacity, and, given it's location and Delaware solid waste management facility siting regulations[6], it is highly unlikely that on-site landfilling capacity could be developed at this site. Furthermore, DuPont has abandoned it's marketing plans for the Iron Rich™ solids in light of its risk assessment of dioxin release in the nearby industrial corridor and has initiated off-site commercial landfilling. Our off-site modeling of this site, therefore, was and is entirely appropriate.

In summary, we disagree with the commenter's assertion that we inappropriately assessed the chloride and chloride-ilmenite processes separately. We believe that for the purposes of the listing determination that our approach provided a sound way to assess the various wastes generated by these processes, and that our approach was applied in a similar way to the two processes. In addition, our assessment of the management practices reported by all of the titanium dioxide manufacturers was reasonable, and remains reasonable in light of the new information provided by the commenter.

Comment 22-2b: EPA's Different And Less Favorable Treatment of Chloride-ilmenite Wastes

When deciding which wastes to evaluate for listing, EPA reviewed the "waste solids" reported by the chloride process plants and determined that the "contribution of any nonexempt solids to the volume of exempt solids . . . would be very small." Thus, the Agency chose "not to attribute any risks to the nonexempt portion of these commingled solids," and did not evaluate those solids for listing. 65 FR 55753. In stark contrast, for the chloride-ilmenite process, EPA separated for analysis many of the non-exempt portions of the exempt-solids streams, and evaluated those non-exempt portions for listing individually and as part of the exempt streams. Thus, EPA proposed to list as K178 non-exempt nonwastewater solids "generated during wastewater treatment and are non-exempt to the extent they are generated from oxidation and finishing wastewaters" and "[c]oke and ore solids ... generated from the removal of solids from waste acid .... if they are removed from the waste acid after the initiation of chemical manufacturing and/or ancillary operations." 65 FR 55760/1. This different and less favorable treatment for solids from chloride-ilmenite operations is not justified anywhere in the proposal, because it cannot be justified.

Similarly, as discussed in Section 3.0 of these comments, EPA improperly evaluated for listing "vanadium wastes" from the chloride-ilmenite process that are not solid wastes, and improperly proposed to list as K178 an unspecified fraction of the coke and ore solids purportedly containing such vanadium wastes, even though the entire coke and ore solids stream is Bevill-exempt. In contrast, EPA did not evaluate "vanadium wastes" from the

---

[6] http://www.dnrec.state.de.us/dnrec2000/Divisions/AWM/hw/sw/swreg.htm

chloride process for potential listing, and did not evaluate for listing coke and ore solids based on similar co-processing of "vanadium wastes" that occurs in the chloride process, as EPA was well aware. (65 FR 55749 and 55765). The wastes generated by the chloride process include: "Vanadium wastes generated in the Purification process" (65 FR 55748/3 and 55749/1). The Agency describes the same stream in the chloride process as follows: "The volatile TiC14, 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. ... Finally, vanadium oxychloride (VOCl3), which has a boiling point close to that of TiC14 (136/C) is removed by complexing with mineral oil and reducing with hydrogen sulfide to VOCl3, or by complexing with copper." See page 6 of Titanium Dioxide Listing Background Document for the Inorganic Chemical Listing Determination, August 2000.

As noted above, EPA provided unjustifiably different treatment by requesting and receiving much less detailed information about the chloride process. As a result, EPA failed to evaluate for listing numerous wastes generated in the chloride process that are equivalent to the wastes EPA evaluated for listing in the chloride-ilmenite process. For example, the Agency states, "Many chloride process facilities commingle waste hydrochloric acid (generated as scrubber water) with their combined wastewaters. Three other facilities, however, return waste acids on site or sell the acids for reuse." 65 FR 55751/3. In addition, the Agency believes that all titanium dioxide manufacturers may generate off-specification titanium dioxide, though this waste was reported only by two of the chloride-ilmenite facilities. 65 FR 55765/3. But even when confronted with evidence that similar streams were produced in the chloride process, such as the "waste acid, which they mingle with coke and ore solids before treatment," EPA refused to evaluate it for listing and simultaneously evaluated similar "waste acid" from the chloride-ilmenite process. 65 FR 55748/2, 55749/1. This inconsistent and arbitrary approach to evaluating wastes from similar processes cannot be justified and is fundamentally unfair.

Finally, EPA's approach to assessing risks from wastes generated by these processes also differed dramatically and without justification. As discussed above, EPA knew or should have known that wastes from the chloride process are managed in similar ways to wastes and products from the chloride-ilmenite process. Nevertheless, EPA performed site-specific risk assessments for wastes from the chloride process while employing excessively conservative generic off-site landfill modeling assessments for wastes from the chloride-ilmenite process. Specifically, based on its knowledge that Iron Rich from DuPont's Edge Moor Delaware facility is sold for off-site beneficial uses involving land application, EPA based its proposed K178 listing on evaluating risks for all non-exempt nonwastewater solids using its off-site industrial non-hazardous landfill model. EPA thus ignored information in the docket demonstrating that non-exempt, nonwastewater solids from DuPont's New Johnsonville Tennessee and DeLisle Mississippi facilities are not managed off-site.

Worse yet, the proposal ignored the site-specific risk assessment that EPA actually performed for New Johnsonville's on-site landfill, which demonstrated that "concentrations of the constituents of concern in the river are likely to be well below the national AWQC for human health and aquatic life for these constituents." 65 FR 55762.

In contrast, for the chloride process, EPA assumed that wastes would continue to be landfilled on-site, and performed site-specific risk assessments that demonstrated the lack of any significant risks. 65 FR 55757 - 55759. As a result, EPA did not propose to list any wastes from the chloride process. However, the residuals generated at DuPont's New Johnsonville and DeLisle facilities are no more likely to be managed off-site than are the wastes generated at chloride process facilities. In fact, DuPont has demonstrated in Section 2.1 that a number of these wastes are being managed off-site. Thus, at a minimum, EPA should not list any wastes from these facilities.

Section 7.0 of these comments demonstrates why EPA should not list any wastes from the chloride-ilmenite process by transferring data and management assumptions from a single DuPont facility and single residual stream to other residuals and facilities, by employing generic and excessively conservative risk modeling, and for numerous other reasons. However, if EPA does mistakenly list wastes from the chloride-ilmenite process, it <u>must</u> also list wastes from the chloride process. [See DuPont's comments and discussion in Section 8.0 regarding conditional and concentration-based listings and an enforceable agreement approach.] Chloride process facilities manage their wastes similarly to chloride-ilmenite process facilities. To avoid arbitrariness, EPA must employ similar management assumptions. This is particularly true given the fact that these processes are really the same and thus should not have been evaluated separately–much less differently–for listing, as discussed below

<u>Response 22-2b:</u> As explained above in response to Comment 22-2a, the listing only impacts solids that are associated with the production of ferric chloride, and thus the listing has no impact on facilities using either the chloride process or the chloride-ilmenite process (and in fact only potentially impacts the DuPont Edge Moor facility). Moreover, EPA's proposal to include all three chloride-ilmenite facilities within its broader listing was reasonable. EPA was aware that the Tennessee and Mississippi facilities managed their solids on-site. EPA, however, reasonably assumed that, if the Delaware facility succeeded in marketing its solids for offsite uses, corporate managers might try the same approach at the other two facilities. The chloride facilities are not owned and managed by the same corporation that owns the three chloride-ilmenite facilities.

<u>Comment 22-2c:</u> The Proposal Mistakenly Differentiates Between Processes That EPA Has Previously Evaluated As The Same Category For RCRA Hazardous Waste Purposes

Until this proposal, EPA repeatedly evaluated the chloride-ilmenite process and the chloride process as a single category for its determinations of which wastes are Bevill-exempt from hazardous waste regulation. Ignoring this long history, the proposal improperly subcategorizes chloride processes of the titanium dioxide industry and evaluates the chloride process and the chloride-ilmenite process separately, using inconsistent approaches. This arbitrary change in EPA's approach is unjustified, and EPA's decision to subcategorize these processes underscores the unfairness of the different and less favorable treatment provided to waste solids from the chloride-ilmenite process.

In the 1989 Bevill Amendment rule, EPA included the wastes generated by DuPont's "chloride-ilmenite" plants in its evaluation of which wastes to retain as exempt mineral processing wastes. The Agency identified DuPont's plant wastes, along with other "chloride process" plant wastes, under a category, "Chloride Processing Waste Solids from Titanium and Titanium Dioxide Production." 54 FR 36593/3 (Sept. 1, 1989). EPA thus "lumped" the chloride process and chloride-ilmenite process wastes into a single category for evaluation, rejecting comments submitted by DuPont that these processes differ and that chloride-ilmenite wastes are beneficiation rather than processing wastes.

In the 1990 Bevill Amendment rule, EPA again considered and rejected DuPont's position that the chloride-ilmenite process and chloride process should be treated separately. "[T]he Agency continues to believe that it is reasonable to consider the chloride-ilmenite process to be a part of the general "chloride process" category for purposes of this rulemaking because the process destroys the identity of the mineral, produces titanium tetrachloride gas (a saleable mineral product), and generates wastes which are functionally identical to, although larger in volume than, the wastes generated by other chloride process facilities." 55 FR 2329/3 (Jan. 23, 1990) (emphasis added). The Agency further stated that "while the description of the chloride process provided in these rules does not describe the ‘chloride-ilmenite’ process in detail . . ., the Agency has clearly considered this process to be one of the several chloride processes covered by these previous rulemakings and, therefore, this rulemaking as well. Id. (emphasis added).

In the January 23, 1990 final rule, EPA justified its classification of DuPont's "chloride-ilmenite" process as "mineral processing" based on the similarity or identity to the chloride process. The primary reason for its determination was that the chloride-ilmenite process "generates wastes which are functionally identical to the wastes generated by the chloride process at the other six titanium tetrachloride facilities." 55 FR 2338/3. EPA further stated that "the fact that the ore being utilized is of a different type and grade is not justification for classifying the operation as beneficiation (versus mineral processing)." 55 FR 2338/3.

In EPA's July 1990 "Report to Congress on Special Wastes from Mineral Processing," EPA reiterated this position. "[T]he slurried residue from the ‘chloride-ilmenite’ process reportedly employed by three titanium tetrachloride production facilities are considered to be chloride process waste solids." Report to Congress, page 13-3.

Even after DuPont successfully sued EPA over this issue, EPA continued to classify the chloride-ilmenite process in the same category as the chloride process. In the May 26, 1998 Phase IV LDR rule (63 FR 28580), EPA concludes, based on its "reexamination of Bevill Exempt Wastes, that “[t]oday's rule is not making any changes to the status of Bevill exempt extraction and beneficiation wastes or the 20 exempt mineral processing wastes." Thus, EPA did not revise its decision that "chloride process waste solids" are Bevill exempt, nor did it establish any regulatory difference between such "waste solids" generated in the chloride process versus the chloride-ilmenite process.

In the May 1998 rule, EPA also states that "[t]he Agency studied the DuPont process numerous times and met with the company several times to assure that the Agency <u>fully understood</u> DuPont [sic] process." (63 FR 28601) (emphasis added)

DuPont submits that the Agency's change in its position in the proposal is inconsistent with its past practice, illogical, and unfair. EPA is evaluating the exact same processes when deciding whether to list the wastes they generate as hazardous. Nothing about EPA's listing approach suggests that differentiating these processes is now warranted. EPA thus should consider the nine facilities producing titanium dioxide as the same "chloride process" category, as the Agency has historically considered them. EPA should then provide uniform evaluations for facilities employing these processes. Consequently, EPA should not list any wastes from any of the <u>nine</u> facilities in the "chloride process" category. If EPA does list any wastes, however, the listing should apply to all similarly situated facilities in this "chloride process" category. [See DuPont's comments and discussion in Section 8.0 (Comment 22-8) regarding conditional and concentration-based listings and an enforceable agreement approach.] As EPA has stated over and over again, there are no differences between these processes or their "functionally identical" wastes that would justify singling out wastes from the chloride-ilmenite process alone for hazardous waste listing.

<u>Response 22-2c:</u> As described above, we believe that our approach to assessing the wastes associated with the three titanium dioxide processes was appropriate and fair. Our court-ordered mandate required that we assess the individual wastes associated with these production processes, and we conducted this assessment in a methodical and efficient manner. The commenter’s concern centers on the perceived inequities of the proposal to list wastes from the chloride-ilmenite process, and to not list wastes from the chloride process (and similarly not from the sulfate process). Our decisionmaking process underlying our proposal, however, considered the actual management practices reported by the industry, and reflected the risks we predicted to be associated with those management practices. Fundamentally, our proposal demonstrated that the chloride process plants manage their wastes onsite, and that these management scenarios posed less risk than the plausible offsite management practices projected for the chloride-ilmenite process.

That being said, however, the commenters have provided substantive comments regarding the scope of the Bevill exemption covering solids from the chloride and chloride-ilmenite processes. As described in response to the following comments, we have found these Bevill-scope comments to be largely persuasive, and have modified the scope of the final rule to reflect the reduced volume of waste that we now believe is not subject to exemption as a special mineral processing waste and continues to warrant listing as a hazardous waste. These solids are associated with the production of ferric chloride acid, and are generated at only one of the nine plants currently manufacturing titanium dioxide.

Comment 22-3: EPA Mistakenly Evaluated In-process Materials For Listing That Are Not Solid Wastes and Mistakenly Proposed to List Bevill-exempt Wastes as Hazardous Wastes

EPA proposes to list as RCRA hazardous wastes (K178) certain "nonwastewaters from the production of titanium dioxide by the chloride-ilmenite process." 65 FR 55748/1 (Sept. 14, 2000). EPA also evaluated for listing "vanadium wastes generated in the purification process," id. at 55749/3, but proposed not to list these "vanadium wastes" based on the lack of risk associated with their management. Id. at 55765/2. Nevertheless, the proposal suggested that an unspecified fraction of certain K178 nonwastewaters - "coke and ore solids removed from the gaseous titanium tetrachloride stream" - contained these "vanadium wastes" and thus were not exempt from hazardous waste regulation under the "Bevill Amendment," RCRA §§ 3001(b)(3) & 8002(p). "We note that any contributions to this [solids] stream from the disposal of the vanadium waste is non-exempt." Id. at 55760/1.

1. DuPont contends that EPA has mistakenly assumed that the so-called "vanadium waste generated in the purification process" is a solid waste. As explained more fully below, DuPont submits that this stream is actually an in-process material, falls outside of EPA's jurisdiction, and should not have been considered as a potential candidate for the proposed RCRA listing. Because this in-process material is not a solid waste, moreover, its further processing is not "disposal" and the coke and ore solids do not contain any disposed "vanadium waste." The entire amount of the coke and ore solids are Bevill exempt. Consequently, the coke and ore solids also should not have been considered as a potential candidate for listing, and they should be excluded from the description of K178 nonwastewaters if any such hazardous waste listing is finalized.

2. DuPont contends that the proposal to include an unspecified fraction of the coke and ore solids in the K178 listing because of "disposal of the vanadium waste" would contradict EPA's prior Bevill determinations. In the past, EPA considered the entire corpus of those solids to be exempt, with full knowledge of the in-process material and its management. The proposal thus contradicts EPA's prior Bevill determinations and EPA's stated intent not to revisit those determinations in this listing.

3. DuPont contends that the proposal also would contradict EPA's prior determinations in the Phase IV Land Disposal Restriction ("LDR") rule. In that rule, EPA determined that co-processing of smaller amounts of mineral processing secondary materials along with Bevill feedstocks would not affect the exempt status of wastes determined to be Bevill exempt. Even if the "vanadium wastes" could properly be considered solid wastes, they would not be "disposed" by co-processing. Thus, all of the coke and ore solids should be Bevill exempt, and EPA should not have considered any of these solids for listing.

Comment 22-3a: EPA mistakenly evaluated the in-process titanium tetrachloride purification stream for listing, because that stream is not a solid waste. Because this stream is not disposed by further processing, EPA also mistakenly proposed to list a fraction of Bevill-exempt coke and ore solids.

Comment 22-3a-1: Overview of titanium tetrachloride process

DuPont and other titanium tetrachloride producers extract titanium from ilmenite or rutile ore by reacting the heated ore with chlorine and coke (a reducing agent) to form titanium tetrachloride. The titanium tetrachloride is then used as a feedstock in production of titanium dioxide. From chlorination of the ore to the beginning of titanium dioxide production, the crude titanium tetrachloride is continuously processed through staged purification steps to maximize yield of the intermediate product, titanium tetrachloride.

Within the process, DuPont and other titanium tetrachloride producers manage various in-process materials towards producing the intermediate product. The first in-process material is crude titanium tetrachloride, which is processed to extract the titanium tetrachloride from un-reacted coke and ore and metal chlorides. The second in-process material is a concentrated titanium tetrachloride slurry ("in-process titanium tetrachloride purification stream"), which is also processed to extract titanium tetrachloride. In processing these materials, a separated un-reacted coke and ore solids matrix is generated.

Comment 22-3a-2: The in-process titanium tetrachloride purification stream is not a solid waste

The proposed rule incorrectly identifies the in-process titanium tetrachloride purification stream as a liquid "non-exempt vanadium waste". 65 FR 55760/5 (Sept. 14, 2000). Rather, this stream is a valuable in-process material that is being further processed to maximize yield of the intermediate product, titanium tetrachloride. This is performed in the most efficient and environmentally friendly manner, by continuously processing the stream within the same process that generates it without any exposure to the environment. In fact, the in-process titanium tetrachloride purification stream is even more valuable than the crude gaseous titanium tetrachloride with which it is co-processed, as it contains significantly higher concentrations of titanium tetrachloride. This stream is not a solid waste, because it is never discarded.

Under RCRA, in order for a material to be considered a "solid waste," that material must first be a "discarded material." RCRA § 1004(27). Congress clearly and unambiguously used the term "discarded" in its ordinary sense to mean "'disposed of,' 'thrown away,' or 'abandoned.'" Congress thus intended "to regulate under RCRA only materials that have truly been discarded." Consequently, materials are not "discarded" and are not "solid wastes" if they are "in-process materials employed in an on-going manufacturing process." Such materials are not being disposed of, thrown away, or abandoned.

American Mining Congress v. EPA, 824 F.2d. 1177, 1184, 1192 (D.C. Circuit 1987) *(AMC I).*

A recent decision confirmed that the decision in AMC I continues to provide the standard for determining when a material becomes a "solid waste." *Association of Battery Recyclers, Inc. v. EPA,* 208 F.3d 1047 (D.C. Circuit 2000) *(ABR).* Even for secondary materials, which the in-process titanium tetrachloride purification streams are not, the court in ABR stated "[T]he secondary material EPA seeks to regulate as solid waste is destined for reuse as part of a continuous industrial process and thus is not abandoned or thrown away. Once again, `by regulating in-process secondary materials, EPA has acted in contravention of Congress' intent. "' *Id*. at 1056 (citing *AMC 1,* 824 F.2d at 1193).

In summary, under the applicable RCRA standards, the in-process titanium tetrachloride purification stream is not a "solid waste." Rather, this material is immediately used within an on-going and continuous process of manufacturing an intermediate product, purified titanium tetrachloride. It is never "'disposed of,' 'thrown away,' or 'abandoned. "' In contrast, it is continuously processed in order to maximize the yield of titanium tetrachloride. DuPont believes, therefore, the in-process titanium tetrachloride purification stream is not within the EPA's jurisdictional authority to regulate under Subtitle C of RCRA.

<u>Response 22-3a-2:</u> See our response to comment 8-4a. We received both supportive and critical comments on our approach to evaluating secondary materials that may be reused or recycled. As discussed in the proposed rule, these determinations are complex, time consuming and best made on a site-specific basis. We continue to believe that this is the appropriate approach and thus, have not made site-specific determinations on whether secondary materials are or are not solid wastes if we could more quickly determine that they did not pose a listable risk.

In the case of the vanadium return stream (also called vanadium recycle stream, and vanadium purge stream), based on our initial analysis, we understood that the vanadium return stream was piped back to the reaction area in totally enclosed piping system and then mixed with other wastes. We determined that the vanadium return stream did not pose a listable risk, as it was managed in totally enclosed piping, until it exited the system and was mixed with other materials (condenser solids). We evaluated the condenser solids elsewhere in the proposal as a separate waste category.

We have since gained a better understanding of how the vanadium return stream is managed and how the condenser solids are generated. We continue to believe that the vanadium return stream is managed in enclosed piping and presents no listable risk and so, we have not evaluated the vanadium return stream further. However, we now understand that the condenser solids and vanadium return stream are not commingled after generation, but instead are generated as a single wastestream and have been so generated for over 20 years. Based on our current understanding of the process configuration, we agree with the commenters, and are modifying our proposed listing approach to view the co-generated condenser solids and vanadium stream solids as a single waste subject to the Bevill exemption at 40 CFR 261.4(b)(7). Since we are not proposing to list this exempt waste, we do not need to determine whether the vanadium return stream is a “solid waste” at any specific point within the process. We do not need to address any issues raised by the Association of Battery Recyclers decision.

Comment 22-3a-3: The proposed rule mistakenly evaluated non-waste for listing and mistakenly proposed to list Bevill-exempt waste

Because the in-process titanium tetrachloride purification stream is not a solid waste, the proposal mistakenly considered whether to list it as a hazardous waste. "Hazardous wastes" by definition must be "solid wastes." RCRA § 1004(5). EPA thus should never have evaluated the in-process stream for listing. Nevertheless, EPA properly proposed not to list this stream as hazardous, as EPA correctly concluded that processing of this stream posed no environmental risk. 65 FR 55765/2. As we describe below, such processing is preferable to alternative means of extracting the titanium tetrachloride from this in-process material.

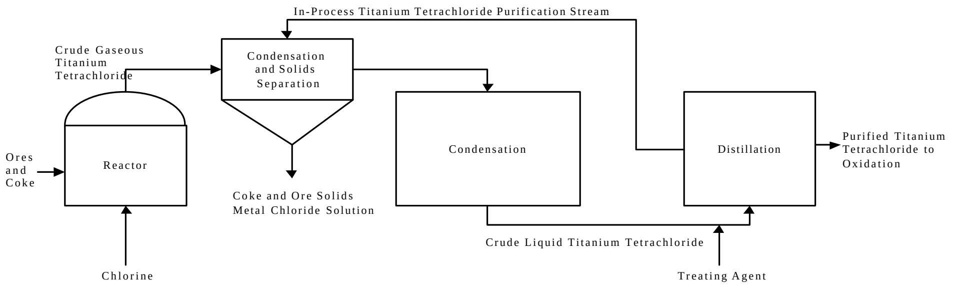
The proposal also mistakenly proposed to list a portion of the Bevill-exempt coke and ore solids matrix, on the belief that it contains non-exempt "disposed" "vanadium waste." Because the in-process titanium tetrachloride purification stream is not a solid waste, it is not "disposed" in the Bevill-exempt coke and ore solids. The drying and separation of solids present in this stream, along with solids in the crude gaseous titanium tetrachloride stream, imparts no effect on the Bevill-exempt status of the chloride process waste solids removed from the titanium tetrachloride process in equipment downstream of the reactor. In the proposal, EPA expressly stated that it was "not reopening any Bevill decisions made in earlier actions regarding the exemptions," and that it would not evaluate for listing exempt streams or "exempt portions" of commingled exempt and non-exempt streams. 65 FR 55688/3. EPA thus should not have evaluated any of the exempt coke and ore solids for potential listing based on the presence of "vanadium waste".

Response 22-3a-3: See our response to comments 22-3a-2 and 8-4a.

Comment 22-3a-4: Diagram and Description of the Titanium Tetrachloride Process

Figure 1 is a depiction of the simplified Chloride-Ilmenite titanium tetrachloride production process employed by DuPont. Because the sequence of steps is believed to be identical, it also depicts the Chloride process employed by DuPont's competitors.

Figure 1 Chloride and Chloride Ilmenite Titanium Tetrachloride Process



As shown in Figure 1 above, in both the chloride-ilmenite and chloride titanium tetrachloride production processes, the chlorination of ilmenite or ruble ores in the presence of coke, which serves as a reducing agent, occurs in a reactor. Hot gas produced in the reactor, which consists of gaseous titanium tetrachloride, other gaseous metal chlorides and un-reacted coke and ore solids, is then cooled and separated in downstream equipment prior to obtaining the intermediate product, purified titanium tetrachloride.

In equipment downstream of the reactor, crude gaseous titanium tetrachloride is extracted from the majority of high boiling metal chlorides and un-reacted coke and ore solids by condensation, drying, and gravity separation. Following this separation, the hot gas is then condensed to obtain a crude liquid titanium tetrachloride. The crude liquid must be further purified to extract titanium tetrachloride from the remaining non-titanium metal chlorides (particularly vanadium chlorides) and remaining suspended solids (e.g., iron chloride and un-reacted coke and ore).

Because vanadium chlorides boil at about the same temperature as titanium tetrachloride, separation is accomplished by distilling the crude liquid titanium tetrachloride in the presence of a treating agent. Distillation results in the production of a gaseous overhead comprised of purified titanium tetrachloride (which is then condensed and used to produce titanium dioxide) and a slurry comprised of approximately 92-97% by weight liquid titanium tetrachloride and 3-8% by weight solids (i.e., vanadium and other metal chlorides, un-reacted coke and ore). This slurry ("in-process titanium tetrachloride purification stream") is then co-processed with hot gas in equipment downstream of the reactor. In this way, titanium tetrachloride is extracted from the in-process titanium tetrachloride purification stream, whereas the solids are dried and separated along with solids in the crude gaseous titanium tetrachloride stream from the reactor.

Response 22-3a-4: See our response to comments 22-3a-2 and 8-4a.

Comment 22-3a-5: Benefits of processing the in-process titanium tetrachloride purification stream

As DuPont explained during its October 26, 2000 meeting with the Agency, the in-process titanium tetrachloride purification stream contains approximately 92-97% titanium tetrachloride, by weight. This accounts for approximately 10% to 20% of all titanium tetrachloride produced by DuPont annually, and translates into a material value of tens of millions of dollars each year. Essentially complete extraction of this material value is accomplished in equipment downstream of the reactor. Furthermore, extracting material values in this manner maximizes yield and significantly reduces the amount of ore required to produce like quantities of the intermediate (purified titanium tetrachloride) and final product (titanium dioxide), conserving natural resources and (as described below) conserving energy.

Continuous processing occurs without any exposure to the environment. The in-process titanium tetrachloride purification stream is co-processed with hot gas that has exited the reactor using enclosed tanks and enclosed piping, which prevents material loss. These in--process materials are not stored or placed on the land, or managed in open units having the potential for releases to the atmosphere. As a result, they pose no risk, as the Agency has acknowledged in the proposed rule. 65 FR 55765 .

Maximizing use of existing process equipment downstream of the reactor to process the in-process titanium tetrachloride purification stream allows DuPont and other titanium tetrachloride producers to transfer and recapture the heat energy in hot gas which has exited the reactor. This saves significant quantities of energy that DuPont and other titanium tetrachloride producers would otherwise have to expend to accomplish similar extraction of titanium tetrachloride in additional equipment. This results in eliminating emissions associated with the fuels that would otherwise have to be burned to operate added equipment and avoids additional capital and maintenance costs of same. DuPont estimates that, for its own facilities, adding to its existing process to accomplish similar extraction of titanium tetrachloride values would translate into tens of millions of dollars net present value. For these reasons, DuPont and all other titanium tetrachloride producers have practiced this preferred, environmentally safe and cost effective method of continuous processing for more than 20 years.

Response 22-3a-5: See our response to comment 22-3a-2.

Comment 22-3a-6: Metal chlorides separated from the in-process titanium tetrachloride purification stream are mineral processing wastes, not chemical processing wastes

The Agency has long maintained "that Congress did not intend the Bevill exclusion to extend to processing operations that are performed *after* the production of a *saleable mineral product* (emphasis added)". 54 FR 36620/1, Sept. 1, 1989. Similarly, the Agency has also maintained that it "considers titanium tetrachloride, produced during the titanium chloride process, to be a saleable [mineral] product; any processing *subsequent* to its production is considered to be chemical processing" (emphasis added). 54 FR 36620/1, Sept. 1, 1989.

The Agency reaffirms that it still maintains this position in its sector specific discussion of titanium dioxide in the proposed rule. "Mineral processing ends when titanium dioxide is produced in the oxidation unit. Further steps are chemical manufacturing. The Agency defines the beginning of oxidation as the beginning of chemical manufacturing because the facility is using a saleable mineral product, titanium tetrachloride, to produce titanium dioxide". 65 FR 55750.

The practice of introducing a treating agent just prior to distillation of *crude* liquid titanium tetrachloride, which is not yet a saleable mineral product, is to facilitate the most economical extraction of the intermediate product, titanium tetrachloride. For this reason, metal chlorides separated from the in-process titanium tetrachloride purification stream are clearly mineral processing wastes, NOT chemical processing wastes.

Response 22-3a-6: See our response to comment 9-2d. Chemical manufacturing, as defined in the September 1, 1989 Bevill rulemaking (54 FR 36592) does not begin with the introduction of a treating agent at the distillation step. Solid wastes from the distillation step would be viewed by the Agency as mineral processing wastes if they are disposed. As noted above, the Agency now acknowledges that vanadium solids separated from ferric chloride waste acid are Bevill exempt mineral processing wastes when disposed.

Comment 22-3b: EPA's proposal to list a fraction of Bevill-exempt coke and ore solids is inconsistent with its prior Bevill determinations and with EPA's stated intent not to revise those determinations in this rulemaking

Comment 22-3b-1: EPA's proposal is inconsistent with EPA's prior Bevill determinations

As noted previously, EPA did not intend to evaluate Bevill-exempt waste for listing and did not intend to revise previous Bevill-exemption determinations in this rulemaking. 65 FR 55688/3. The proposal, however, does just that by evaluating for listing an unspecified fraction of the coke and ore solids, which EPA claims to be not Bevill-exempt due to the "disposal" of "vanadium waste." This statement is inconsistent with the long history of EPA's extremely careful scrutiny of DuPont's chloride-ilmenite process and the wastes generated by that process for purposes of determining application of the Bevill Amendment. Throughout those evaluations, EPA was fully aware that DuPont co-processes the in-process titanium tetrachloride purification stream. EPA never suggested, until this proposal, that such co-processing would adversely affect the status of the coke and ore solids that EPA determined were Bevill-exempt.

It also bears noting that EPA subjected DuPont's process to as much, if not more, exacting scrutiny than any other process evaluated in EPA's Bevill determinations. EPA evaluated DuPont's Chloride-Ilmenite process and associated wastes beginning in the early 1980s and leading up to its initial decisions in 1989, revisited these decisions in 1990 in a final rule and Report to Congress, then reconsidered these decisions in its 1991 Final Regulatory Determination, considered DuPont's process yet again during litigation in 1991 and 1992, reevaluated them in 1996 in response to that litigation, and reached final conclusions in 1998. Unlike for other processes, where EPA may have made general determinations without serious scrutiny of the affected processes, EPA cannot claim that it is simply "determining whether particular wastestreams fall within any of the exempt categories." 65 FR 55688/3. Instead, the proposal in fact is "reopening ... earlier decisions as to which categories of mineral processing wastes are exempt" and is revising prior decisions regarding the exempt status of particular wastestreams.

Response 22-3b-1: See our response to comment 22-3a-2. As noted above, the Agency now acknowledges that the solids containing vanadium, separated from ferric chloride waste acid, are Bevill exempt mineral processing wastes when disposed.

Comment 22-3b-2: EPA was fully aware that DuPont co-processed the in-process titanium tetrachloride purification stream and never suggested, until now, that such processing would adversely affect the Bevill-exempt status of the coke and ore solids.

EPA carefully evaluated DuPont's chloride-ilmenite process in the 1980s in order to make its initial Bevill determinations. Following those determinations and contemporaneous with the court ruling in Solite Corp v. EPA, 952 F.2d 473, 494-95 (D.C. Cir. 1991), EPA issued a RCRA § 3007 questionnaire that documents EPA's knowledge of DuPont's process gleaned from its prior evaluations. That questionnaire expressly records EPA's understanding that DuPont co-processed the in-process titanium tetrachloride purification stream. However, EPA did not suggest then or at any other time until the current listing proposal that such co-processing adversely affected the Bevill-exempt status of a fraction of the coke and ore solids.

1990 Report To Congress

EPA's July 1990 "Report to Congress on Special Wastes from Mineral Processing" (EPA/530-SW-90-070C) also demonstrates that the Agency was fully aware of DuPont's operations and that it considered all of the coke and ore solids to be Bevill-exempt "chloride process waste solids." In the 1990 Report, the agency devotes a Chapter to Titanium Tetrachloride Production - Chapter 13. EPA provides Exhibit 13-2, a simplified process flow diagram of "titanium tetrachloride production." In this diagram, EPA identifies a process step as "chlorination/purification" in which titanium tetrachloride and waste acids and solids exit.

The titanium tetrachloride goes to oxidation or reduction to produce titanium dioxide or titanium sponge. The waste streams, however, are identified as proceeding to neutralization and solids/liquid separation. Up to this point, EPA describes these unit operations as the "process." However, EPA indicates that exiting the neutralization or solids/liquids separation step are the "chloride process waste solids." This "chloride process waste solids" step is also identified as the "special waste management" or materials that are considered to be "Bevill-exempt" under RCRA.

EPA developed Exhibit 13-2 in its Report to Congress based on a "number of data collection activities" and described how "the focus of most of these efforts was site-specific." Therefore, EPA concluded that it had been able to "compile detailed facility- and sector-specific information." (Report to Congress, page 2-1)

EPA also reported on its 1989 National Survey of Solid Wastes from Mineral Processing Facilities in which questionnaires with about 300 questions were sent to about 200 facilities. EPA states that "facility operator responses to the questionnaire provide <u>nearly complete coverage</u> of the facilities that currently generate one or more of the 20 special study wastes." (emphasis added) (page 2-1) EPA further states that "coverage for many of the 20 waste streams is complete. . ."

In Appendix B-1 of the Report to Congress, EPA described the purpose of the 1989 National Survey of Solid Wastes as a need "to obtain information that <u>specifically</u> pertained to the facilities, processes, and management practices that are associated with the ore and mineral processing wastes that are covered by the Mining Waste Exclusion." (page B-1) The Agency then describes certain sections of the Survey questionnaire, including Section 2 - Processing Units That Generate a Special Waste. EPA indicates that the questions in this section pertain "to the specific points in the production process at which the special wastes were and are generated." The Agency further states that "the emphasis of the section was on gaining knowledge of how, where, and why these materials are generated," and that "respondents were asked to describe all on-site processes that generate each waste of concern."

As identified in Appendix B-2 of the Report to Congress, EPA indicates that 37 facilities were selected for sampling, including DuPont's DeLisle (Pass Christian), MS and DuPont's Edge Moor, DE plant. (See Exhibit B-2-1) (page B-2-5) Therefore, based on EPA's own description, it visited and obtained samples from two of DuPont's plants and received information regarding the production processes at which special wastes were and are generated. Thus, based on the information at hand from the 1989 survey responses and its own visits to DuPont's plants, it seems inconceivable that EPA was not fully aware of DuPont's in-process titanium tetrachloride purification stream at two of DuPont's plants. Yet, EPA made no statements until nearly a decade later, during the subject proposed listing, that this stream would be considered a waste stream, potentially subject to Subtitle C of RCRA, and would cause a fraction of the coke and ore solids to lose their exempt status.

In its narrative description in the Report to Congress, EPA states that "the nonvolatile chlorides and the unreacted process solids that remain after the reaction in the fluidized-bed reactor (or chlorinator) are the special waste under study in this report. These solids, suspended in chloride process waste acids, are treated and discharged." EPA goes on to say that "as noted in the January 23, 1990 final rule (54 FR 2322), the slurried residue from the 'chloride-ilmenite' process reportedly employed by three titanium tetrachloride production facilities (DuPont's plants) are considered to be chloride process waste solids."

Based on EPA's own Exhibit 13-2, the agency considered that "chloride process waste solids" generated from the "chlorination," as well as the "purification" step, are considered to be Bevill exempt solids.

1992 RCRA Section 3007 Questionnaire

DuPont received on November 17, 1992 a request for information, pursuant to RCRA Section 3007, from the Chief, RCRA Compliance Section in EPA Region IV, containing 18 very detailed and specific questions regarding the DuPont Johnsonville, TN plant's "chloride-ilmenite" process. This request clearly reflects EPA's full knowledge that DuPont was co-processing the in-process titanium tetrachloride purification stream.

EPA's letter explained that "The United States Environmental Protection Agency (EPA) is investigating the extent and nature of the transportation and disposal of hazardous substances or hazardous wastes at DuPont's Johnsonville Plant, located in New Johnsonville, Tennessee. . ." Questions 16 and 17 from this RCRA 3007 information request specifically address the in-process titanium tetrachloride purification stream. EPA's questions and DuPont's responses are presented below:

> "EPA Q16. Describe in detail the process in which titanium and/or titanium tetrachloride and/or titanium dioxide is separated from the vanadium waste stream."
>
> DuPont Response: "In purification, crude TiC14 is treated with an agent which converts vanadium chloride impurities to insoluble solids. After distillation (evaporation and condensation), pure TiC14 is pumped to oxidation.
>
> About 0.6% insoluble vanadium-containing solids in TiC14 with other chlorides which do not evaporate are returned to the spray condenser where the remaining TiC14 is recovered."
>
> "EPA Q 17. State how much titanium and/or titanium tetrachloride and/or titanium dioxide is recovered from the vanadium waste stream when it is recycled. Include in your answers a detailed description and/or analysis of the vanadium waste stream before it is recycled."

> DuPont Response: “The composition of the vanadium containing slurry, 98.9% TiC14, is given in . . . in answer to Question 8 and 12. All of the TiC14 in this stream .... is recovered in the spray condenser where it is vaporized."

The above DuPont responses to EPA's detailed 1992 questionnaire further demonstrates that EPA was again informed in 1992 that the in-process titanium tetrachloride purification stream from purification was recirculated to the spray condenser to further process titanium tetrachloride for DuPont's titanium dioxide plants.

1998 Phase IV LDR Rule Reevaluation

Notwithstanding receipt of this questionnaire [and DuPont's Comments,] EPA issued a final determination in 1998 regarding DuPont's process that never once suggested that co-processing of the "vanadium waste" stream caused a loss of Bevill-exempt status for a portion of the coke and ore solids. In fact, EPA was required by Solite to re-evaluate whether "iron chloride waste acid" was a beneficiation or mineral processing waste. But that acid also derived from co-processing of the in-process titanium tetrachloride purification stream. Consequently, had EPA then believed that "vanadium waste" was disposed of in coke and ore solids by co-processing, it would also be disposed of in the iron chloride waste acid. Yet EPA did not then suggest that a portion of the "waste" under evaluation was already non-exempt for this reason, and thus did not require any evaluation.

In summary, EPA was continuously aware of the co-processing of DuPont's in-process titanium tetrachloride purification stream. Nevertheless, EPA concluded that the coke and ore solids were Bevill exempt - and the iron chloride acid waste was not Bevill exempt - in their entirety. At no point in any of these prior determinations did EPA suggest that some portion of the "downstream" wastes were not exempt due to disposal of "vanadium waste." EPA's suggestion to this effect in the proposal thus is inconsistent with those prior determinations.

Response 22-3b-2: See our response to comment 22-3a-2. The Agency has carefully reviewed its record regarding its understanding of how vanadium compounds are generated during the production of titanium tetrachloride. In 1990, the Agency concluded that vanadium compounds were generated as a distinct separate waste stream. The Agency acknowledges that DuPont did in fact provide information on how vanadium compounds were generated at their facilities in response to a 1992 Agency inquiry. The Agency can only conclude that this process information was not adequately reviewed at the time, and was not relied upon when the Agency issued its draft 1996 Identification and Description of Mineral Processing Sectors and Waste Streams document. In 1996, the Agency also issued a draft document which did reevaluate the generation of ferric chloride waste acids, but this evaluation also did not specifically review if there was a link between vanadium wastes and ferric chloride waste acid generation. It was not until the Agency was informed by DuPont in comment on the draft of this rule, that the Agency was able to fully evaluate how vanadium compounds are generated. The Agency acknowledges that its understanding of how vanadium compounds are generated was incorrect.

As explained in the proposal, we consider the solids from the initial reaction of coke and ore which are separated from the gaseous product stream in the condenser unit to be Bevill-exempt. However, at the time of proposal we thought that facilities commingled these exempt solids after they had been removed from the process with a separate, non-exempt waste stream containing vanadium impurities (generated during titanium tetrachloride purification). We thought gaseous titanium tetrachloride was recovered from this mixture of commingled wastes and returned to the process, and that solid materials, consisting of the condenser coke and ore solids, as well as the non-titanium tetrachloride portion of the vanadium impurities stream, remained outside the process and were ultimately disposed of as a waste. We thought that the remaining portion of the vanadium impurities stream was commingled with the coke and ore solids after those solids were removed from the condenser. We proposed that these separate solids derived from the vanadium impurities stream would be covered by the K178 listing.

We now understand that the residuals from the vanadium impurities stream leaves the process as an integral component of the coke and ore solids. Consequently, we no longer consider the vanadium impurities stream to be a separate waste. Rather, they are an integral component of the coke and ore solids.

For these reasons, we have decided to modify our proposed position on the Bevill status of the vanadium impurities stream. The residuals that exit the condenser are part of the solids from the production of titanium tetrachloride exempt under 40 CFR 261.4(b)(7)(S). This supersedes all earlier positions expressed on the Bevill status of the vanadium impurities stream as we now are aware that we previously misunderstood the details of the process.

Comment 22-3b-3: EPA's proposal to list a fraction of Bevill-exempt ore and coke solids is inconsistent with its position on co-processing in its May 26, 1998 Phase IV LDR Rule.

Comment 22-3b-3-1: Even if the in-process titanium tetrachloride purification stream were considered a solid waste, co-processing it would not affect the Bevill-exempt status of the coke and ore solids

EPA addresses the reclamation of mineral processing secondary materials in the May 26, 1998 final Phase IV LDR rule. In that rule, the Agency decided that any environmental concerns associated with co-processing of mineral processing secondary materials with normal raw materials were outweighed by the environmental benefits of such recycling. In particular, EPA was "concerned that [imposing a "significantly affected" test for determining when such co-processing would alter the Bevill-exempt status of wastes] would severely disrupt legitimate recycling practices within beneficiation and mineral processing industries." 63 FR 28578/3 (emphasis added). EPA finally concluded that:

> Today's rule also allows secondary materials from mineral processing to be coprocessed with normal raw materials in beneficiation operations (and presumably

> mineral processing operations based on statements cited above) which generate Bevill exempt wastes, without changing the exempt status of the resulting Bevill waste, provided that legitimate recovery of the mineral processing secondary material is occurring, and provided that primary ores and minerals account for at least 50 percent of the feedstock. 63 FR 28578.

Therefore, based on EPA's final Phase IV LDR rule, even if the in-process titanium tetrachloride purification stream were considered a "solid waste" stream, which DuPont contends it is not, then the co-processing of this in-process stream, with other Bevill raw materials in the spray condenser, would not remove the Bevill-exempt status of the entire volume of coke and ore solids. Nor would it result in the "disposal" of "vanadium waste" in those solids, subjecting them to the "Bevill mixture-rule." The coke and ore solids would continue to be considered Bevill exempt because the in-process titanium tetrachloride purification stream is far less than 50% of the total feed into the spray condenser where the titanium tetrachloride is extracted.

Because co-processing does not affect the Bevill-exempt status of the coke and ore solids, EPA should not have proposed any fraction of those solids for listing

Because EPA did not intend to list wastes that are Bevill-exempt, it should not have proposed to list a fraction of the coke and ore solids. These solids would remain Bevill-exempt even if the in-process titanium tetrachloride purification stream were considered a solid "vanadium waste." Further, EPA should exclude from any final K178 listing all coke and ore solids generated by co-processing of this in-process stream.

Response 22-3b-3-1: The Agency's LDR Phase IV rule evaluated whether the introduction of legitimately recycled materials would adversely affect the Bevill status of mineral processing facilities. The Agency's proposed rule sought comment on whether a particular stream which may have been composed of both a legitimate recycled material and a waste would be regulated from a Bevill and listing standpoint. As noted earlier, this issue is now moot since the Agency has found that solids from the vanadium impurities stream that exit the condenser along with coke and ore solids are exempt mineral processing wastes. EPA would regard the vanadium solids as exempt even if the vanadium return stream was a waste before it exited the condenser along with the coke and ore solids.

Comment 22-4: EPA Mistakenly Proposed For Listing Ferric Chloride Solids Because These Solids Are Not Produced as The Result of Chemical Manufacturing And/or Ancillary Operations Nor Are They Subjected to Chemical Processing That Alters the Properties of the Solids

EPA proposes to list as RCRA hazardous wastes (K178) certain "nonwastewaters from the production of titanium-dioxide by the chloride-ilmenite process." 65 FR 55748/1 (Sept. 14, 2000). As part of this listing, EPA proposed to include ferric chloride solids from DuPont's

Edge Moor, Delaware facility, which the Agency believes are produced while adding a chemical additive to ferric chloride in a chemical manufacturing process. Specifically, the proposal states that "at the Delaware facility, solids that collect in the ferric chloride product storage tanks and impoundments would be covered by the listing as these solids are ineligible for the mineral processing exemption (because they are generated after the initiation of chemical manufacturing and/or ancillary operations)" 65 FR 55763/2

1) DuPont contends that in the September, 14, 2000 proposed listing, EPA has mistakenly assumed that the addition of chlorine to the ferric chloride stream from the titanium tetrachloride process generates or affects the unreacted coke and ore solids that are separated from that solution. However, these solids have already been separated from titanium tetrachloride in the titanium dioxide production process, are carried along with the "waste acid" through the point of chlorine injection, are not affected by the chlorine injection, and are Bevill-exempt whether separated from the ferric chloride solution before or after chlorine injection. As the Agency recognizes, these solids are exempt when separated before addition of chlorine gas. The Agency should recognize that they retain their same character and exempt status after the addition of chlorine.

2) DuPont contends that the proposal to include the solids from the ferric chloride, which collect in the Iron Rich^{TM}, ferric chloride product storage tanks and impoundments, would contradict the EPA's prior Bevill determinations. During prior Bevill determinations, EPA sampled these solids and agreed that they were exempt. The exempt status of these solids was understood not only by the facility and EPA, but also by the regulating state agency.

3) DuPont contends that the proposal also would contradict the Agency's standards for distinguishing mineral processing from chemical manufacturing, 54 FR 36592, 36616 (Sept. 1, 1989), and its clarification of "uniquely associated" wastes in the Phase IV Land Disposal Restriction ("LDR") rule. The solids when disposed of are solid wastes that are uniquely associated with and originate from mineral processing operations.

4) Because the solids separated from the ferric chloride solution at Edge Moor are Bevill-exempt, EPA should not have evaluated them for listing. If EPA issues any final K178 listing, it should reiterate that these solids are Bevill-exempt and should exclude them from the scope of the listing. The Agency should acknowledge that any solids separated prior to or after chlorine injection are Bevill-exempt and should exclude those solids from any K178 listing.

Comment 22-4a: Overview of the Ferric Chloride Process and Mistaken Belief That Solids Are Generated by Addition of Chlorine

The Delaware site has produced Ferric Chloride since 1974 using chlorine to convert remaining levels of ferrous chloride to ferric chloride and extract the full value of the original mineral matrix. The same location has been used since 1974. The technology of adding chlorine to the in-process stream exiting the equipment downstream of the titanium tetrachloride reactor, which is called "trim chlorination," effectively turned what would have become a "waste acid" into a valuable commodity used for water and wastewater treatment.

To produce this saleable product, small amounts of gaseous chlorine are added at low temperatures (< 100C) to a ferric chloride solution containing small amounts of ferrous chloride (3%). The chlorine is added to convert the ferrous iron to ferric iron. Chlorine addition is carefully metered in stoichiometric amounts.

The solids that are separated from the ferric chloride solution consist of unreacted coke and ore from the titanium tetrachloride reactor. These solids are inert, and thus are essentially unaffected by the addition of chlorine to the ferric chloride stream. They pass through the ferrous-to-ferric reaction zone without any change in their chemical composition or quantity.

DuPont has practiced this technology to produce ferric chloride acid product since 1974. DuPont has never considered the addition of small amounts of chlorine to be chemical processing, but rather an integral part of its mineral processing operations. The small amount of chlorine allows DuPont to extract iron value from the original ore matrix, by converting the ferric chloride to a commodity chemical, rather then creating a "waste acid" that would require treatment and disposal.

As the Agency recognized in the proposal, the solids in the ferric chloride solution are Bevill-exempt once they are separated by filtration from the solution at DuPont's DeLisle Mississippi and New Johnsonville Tennessee facilities. 65 FR 55759/3. At those plants, the separated ferric chloride solution is disposed by deepwell injection and is used as a feedstock in sodium chloride production, respectively. At DuPont's Edge Moor Delaware facility, the ferric chloride solution is sold as a product after separating the solids. The only significant difference in the generation of these solids at Edge Moor is the addition of chlorine prior to separation. DuPont does not believe that this method of enhancing its recovery of usable iron values warrants a change in the Bevill-status of the solids at Edge Moor. As discussed above, this additional step does not in any way affect or alter the nature of the solids generated.

Consequently, DuPont does not agree with the proposal that the addition of chlorine causes the solids to be generated from a non-exempt chemical processing operation. Id., 65 FR 55760/2. (DuPont also does not agree that any portion of these solids contain nonexempt "vanadium waste," as discussed in Section 3.0 of these comments. See id.) As demonstrated below, by treating the solids separated from the ferric chloride solution as not Bevill-exempt, the proposal is inconsistent with EPA's prior determinations that this specific stream is Bevill-exempt and with EPA's standards for determining what wastes are generated from mineral processing rather than chemical manufacturing.

Response 22-4a: We disagree with these comments. We believe that wastes from the production of ferric chloride are not wastes that are exempt under the Bevill exemption regulations. They are not extraction and beneficiation wastes because the input material (waste acid containing solids from the titanium dioxide manufacturing process) has gone through mineral processing. Once mineral processing begins, all subsequent operations are not considered extraction or beneficiation. See 54 FR at 36619, September 1, 1989. Even if they were considered mineral processing wastes, they are not wastes from any of the 20 specific mineral processing wastes exempted under 261.4(b)(7)(ii). As explained in the proposal, we believe that once the Delaware facility adds chlorine to the waste acid stream, it is engaged in the manufacture of ferric chloride, not the manufacture of titanium tetrachloride, the material for which wastes are exempt under 261.4(b)(7)(ii)(S).

In support of our position, we note that the manufacture of ferric chloride is in no way necessary to the manufacture of titanium tetrachloride or titanium dioxide. The facility does not use any of the ferric chloride in any step of the process that produces either of the two titanium products.

Comment 22-4b: The Proposal Is Inconsistent With EPA's Prior Determinations That The Solids Separated From Ferric Chloride Solution At Edge Moor Are Bevill-Exempt

When making its Bevill-determinations for chloride-ilmenite mineral processing wastes, EPA carefully reviewed and was fully aware of DuPont's process at the Edge Moor facility. (A more complete discussion of the history of EPA's Bevill analyses is provided in Section 1.0 above.) Based on its review, EPA listed solids from DuPont's streams without excluding the solids generated from ferric chloride solution at DuPont's Edge Moor facility. See 40 CFR § 261.4(b)(7). This decision that the ferric chloride solution solids were Bevill-exempt was consistently reaffirmed through the 1989 "National Survey of Solid Wastes from Mineral Processing," the 1990 Bevill determination, the 1990 Report to Congress on Special Wastes from Mineral Processing, and the 1998 Phase IV LDR Rule, where the Agency reevaluated the Bevill status of the ferric chloride solution. Only in this proposal has EPA's evaluation changed. The proposal is therefore inconsistent with EPA's prior decisions, and contrary to the Agency's claim that it is "not reopening any Bevill decisions made in earlier actions regarding the exemptions." 65 FR 55688/3.

Significantly, when making its prior decisions that ferric chloride solution solids from all of DuPont's facilities were Bevill exempt, the Agency actually sampled and analyzed the solids it now claims are not exempt. In Chapter 13, page 4 of EPA's 1990 Report to Congress, EPA states that it analyzed 16 samples from among the nine U.S. titanium tetrachloride production facilities. DuPont's Edge Moor facility was one of the facilities from which chloride process waste solids were sampled. The location of the waste solids sample was from an impoundment. These waste solids included solids separated from the ferric chloride stream after addition of chlorine. Thus, there can be no mistake that EPA's prior Bevill determinations reached this issue and concluded that the trim chlorination solids at Edge Moor were Bevill-exempt.

Response 22-4b: The commenter observes that we sampled the waste solids from the production of ferric chloride in the mid-1980's, and, when we established the exemption for solid titanium tetrachloride wastes in 1991, we did not assert that these solids were not covered by the exemption. The commenter may be correct that our mid-1980's sample of commingled solids included some solids filtered out of ferric chloride production. However, we did not know, at the time that we promulgated the titanium tetrachloride exemption, that the plant filtered out the solids after it added chlorine to the waste acid (i.e., began the manufacture of ferric chloride). The regulatory language, however, is sufficiently clear: EPA defined the exemption as applying to solids from the manufacture of titanium tetrachloride, not ferric chloride production. See Response 12-2a(1) for EPA's response on re-opening Bevill issues.

Comment 22-4c: The Proposal Is Inconsistent With EPA's Existing Standards For Determining Bevill-Exempt Status

When making its Bevill determinations in 1989, EPA articulated three criteria for determining when mineral processing ends and when chemical manufacturing begins. 54 FR 36614 (Sept. 1, 1989). To qualify as mineral processing wastes, these criteria require that:

(1) The material must be a solid waste as defined by EPA; (2) The solid waste must be uniquely associated with mineral industry operations; and (3) The solid waste must originate from mineral processing operations.

Because the solids separated after addition of chlorine to the ferric chloride solution at DuPont's Edge Moor facility meet all 3 criteria, they are exempt mineral processing wastes when disposed. (This is the reason that EPA did not exclude the Edge Moor solids from its prior Bevill-exemption determinations for the chloride-ilmenite process.)

EPA's criteria are met because: (1) the solids are solid wastes as defined by EPA when they are disposed, (2) the solids are uniquely associated with, and (3) originate from the mineral processing operations. The waste solids are not formed or created from an ancillary process, but rather from the beneficiation and processing of ilmenite ore. The solids that are removed from the ferric chloride and the solids that collect in the ferric chloride storage tanks and impoundments originate from these mineral processing operations, as they are composed of unreacted coke and ore used in those operations. These solids are merely entrained in the ferric chloride. They are in no way produced as a result of any chemical manufacturing of ferric chloride, and they do not undergo any change as a result of chlorine being injected into the pipeline prior to their removal. Nor does the addition of a coagulant added for the purpose of separating the solids affect their status as Bevill-exempt, as the Agency has recognized at DuPont's other facilities.

EPA's own documents support this understanding that the solids are simply entrained in the ferric chloride, and are not generated by any chemical manufacturing process. The 1990 Report to Congress at page 13-3, states:

The non-volatile chlorides and the unreacted process solids that remain after the reaction in the fluidized-bed reactor are the special waste under study in this report. These solids, suspended in chloride process waste acids, are treated and discharged. As noted in the January 23, 1990 final rule (54 FR 2322), the slurried residue from the "chloride ilmenite" process reportedly employed by three titanium tetrachloride production facilities are considered to be chloride process waste solids.

Response 22-4c: We disagree. We agree with the commenter that the ferric chloride residues are “solid wastes” under the first criterion. However, the waste ferric chloride residues do not meet the second criterion. For a waste to be “uniquely associated” with the titanium tetrachloride mineral processing operation, the process that generates the waste must be necessary to the production of titanium tetrachloride. As explained above, the Delaware plant does not need to make ferric chloride to manufacture titanium tetrachloride, the only material produced there that gives rise to Bevill-exempt wastes. The plant uses no portion of the ferric chloride produced. Since the ferric chloride residues fail to meet this criterion, we have no need to determine whether they meet the third criterion. Moreover, we would take the position that the ferric chloride residues were not exempt even if we agreed that they “originated” in the production of titanium tetrachloride. Residues removed after the facility begins the manufacture of the distinct ferric chloride product (by the addition of chlorine) are not solids from the manufacture of titanium tetrachloride.

Comment 22-4d: Because The Proposed Rule Mistakenly Proposed to List Bevill-Exempt Solids, The Final Rule Should Reaffirm The Bevill-Exempt Status of the Solids And Should Exclude Those Solids From Any K178 Listing Description

Because the unreacted coke and ore solids in the ferric chloride are not created or modified by the addition of chlorine, but rather are generated from mineral processing operations, they are Bevill exempt. The proposal thus mistakenly considered whether to list as K178 the solids separated from the ferric chloride solution after trim chlorination at DuPont's Edge Moor facility. Under its stated policy of not evaluating Bevill-exempt wastes for listing, because they are not within the scope of the consent decree, see 65 FR 55687, 55688, EPA should never have evaluated these solids in the proposal. When EPA issues its final rule, it should therefore clarify that these solids remain Bevill-exempt and it should exclude these solids from any final K178 hazardous waste listing description.

Response 22-4d: The commenter asserts that the addition of chlorine in the process of making ferric chloride does not alter the solids in the waste acid that it later filters out and mingles with all of its other process solids. This is irrelevant. The issue for the purpose of the Bevill exemption is whether the facility is making titanium tetrachloride or some other product. In determining whether a waste falls within the scope of Bevill exemption for one of the 20 mineral processing wastes, we have never engaged in extending the Bevill applicability to the production of a different product based on an analysis of the similarities or dissimilarities of the waste material.

Moreover, we disagree with this assertion. The waste matrix of concern contains both solids and a measurable amount of liquid waste acid. While we are not convinced that the solids are unaffected by the addition of chlorine, clearly the liquid acid portion of the waste solids has been chemically altered by the addition of chlorine (i.e., the purpose of the chlorine addition is to shift the balance between ferrous and ferric chloride in the acid[7]). Therefore, we believe that at a minimum the acid component of the ferric chloride residue waste matrix does undergo some chemical change as a result of the ferric chloride manufacturing process. Therefore, we do not conclude that the ferric chloride solids are exempt under the Bevill exemptions.

Comment 22-4e: Solids That are Generated Off-site from Ferric Chloride Should not be Included in the Listing

Solids that are generated off-site from the ferric chloride product used at or sold from DuPont's Edge Moor facility should be excluded from the scope of the listing description. Because such solids have essentially the same chemical composition as the Bevill-exempt solids, they should not lose their Bevill-exempt status. The ferric chloride acid is a legitimate product and EPA should not create confusion for users or discourage its purchase and use by applying the listing broadly to the small quantity of sedimented solids generated by any person who stores or uses this acid. It is DuPont's understanding, based on discussions with the Agency, that EPA does not intend for the proposed K178 listing to apply to facilities beyond the boundaries of the chloride ilmenite titanium dioxide production facilities. If the Agency persists in listing the ferric chloride solids, it should clearly allow for an exclusion of the solids that settle out off-site in customer tanks and equipment similar to "Definition of Solid Waste Exclusions" 63 FR 42118 and "Headworks Exemption" 63 FR 42120 afforded the petroleum refining sector.

Response 22-4e: We did not propose to regulate as listed hazardous waste those solids generated off-site from the use of the DuPont Edge Moor's ferric chloride acid. We did not collect data on the generation of these solids, and did not intend to incorporate such solids within the scope of the listing. Users of ferric chloride (e.g., for wastewater and water treatment) have access to a variety of sources of ferric chloride. For example, the major source of ferric chloride is from steel pickling liquor.[8] To the extent that ferric chloride users purchase and store ferric chloride from various venders, the solids settled from the commingled acids may have different properties and compositions than solids entirely associated with DuPont Edge Moor's acid. Any determination on ferric chloride solids from multiple sources is well outside the scope of the inorganic chemical listing determination and the directions provided by the EDF consent decree.

We note, however, that such solids are clearly not eligible for exemption as special mineral

---

[7] See section 4.1 of DuPont's November 13, 2000 comments, as well as letter dated May 4, 2001 to Lillian Bagus, EPA, and Stephen Hoffman, EPA, from Gregg Martin, DuPont, regarding "Proposed K178 Hazardous Waste Listing of Ferric Chloride Solids".

[8] http://www.chemexpo.com/news/PROFILE001127.cfm.

processing wastes. As described earlier in response 22-4c, production of ferric chloride marks the initiation of chemical manufacturing, and solids subsequently generated cannot be special mineral processing wastes. These solids, when generated off-site, would be hazardous waste if they exhibit any of the hazardous waste characteristics.

Comment 22-5: EPA Should Revise its Proposed Listing to Drop Thallium as a Listing Basis and to Exclude Wastes That Do Not Clearly Meet the Listing Basis.

Comment 22-5a: Thallium should not be included in the listing basis for K178.

EPA's proposed listing for thallium in the K178 proposal is based on an SPLP value for thallium for Iron RichTM from the Delaware site in Table III-54 (65 FR 55761) of the proposal. Table III-54 presents the following thallium data for Iron Rich™ (SAIC designation DPE-SO-O1) and for the Tennessee site sludge as follows:

	Total (mg/kg)	TCLP (mg/L)	SPLP (mg/L)
Delaware	3.7	0.28	0.012
Tennessee	7.2	not reported	<0.0022

Comment 22-5a-1: EPA's thallium TCLP value for Iron Rich™ is artificially high.

The above Delaware site data come from SAIC's "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 facility. Table 4-2 of the SAIC report footnotes the corresponding Delaware site thallium TCLP result with the following statement:

> Listed results are less than the typical laboratory reporting limit, but are greater than the calculated instrument detection limits. Concentrations reported in this manner were necessary in order to correlate with the health-based risk assessment limits.

This suggests that thallium was actually found in the TCLP extract, albeit at a concentration below the typical laboratory reporting limit. For the above data, the calculated ratio of the total value to the TCLP value is 13.2. In other words, the maximum theoretical thallium TCLP concentration based on a total thallium concentration of 3.7 mg/kg would be 0.185 mg/L.

Since Section 2.2 of SW-846 Method 1311 for TCLP indicates that the "amount of TCLP extraction fluid is equal to 20 times the weight of the solid phase", the ratio of total to TCLP should have been at least 20. Therefore, the reported TCLP value for thallium for Iron Rich™ solids is not logical.

Additionally, in response to DuPont's request for the APPL lab report and raw data supporting the SAIC Report, EPA provided the raw instrument output from this particular ICP analysis. Review of these raw data by independent data validator Environmental Standards, Inc. indicates that the TCLP result for thallium is likely to be the result of contamination because it is less than 5 times the associated continuing calibration blank (CCB) result. See item 2 of the Environmental Standards letter (Blye to Timmons, Nov. 10, 2000) in Attachment 5.1.1-A

Response 22-5a-1: To consider the validity of DuPont's criticism of our analysis for thallium, we re-examined our total, TCLP and SPLP analyses for thallium.

We first examined the laboratory reporting limit for TCLP thallium analysis. The thallium detection limit for aqueous samples is listed as 5 ug/l in the Quality Assurance Project Plan (QAPP) associated with the Inorganic Chemicals Listing Determination. The respective TCLP procedure was completed using SW-846 Methods 1311, the leachate was extracted according to Method 6010B followed by analysis with an ICAP-61E Trace Level Analyzer. The TCLP leachate was diluted by a factor of 20 prior to extraction to compensate for the high level of sodium contained in the acetate buffer leaching solution. After considering the leachate dilution factor of 20 multiplied by 5 ug/l, the TCLP thallium actual reporting limit should have been 0.1 mg/l. After further discussions with the laboratory, we confirmed that the actual TCLP thallium reporting limit is 0.1 mg/l based on the dilution factor correction. Therefore, the initial laboratory reported TCLP thallium result for DPE-SO-01 of < 2 mg/l was later corrected to 0.28 mg/l, which is above the laboratory TCLP thallium reporting limit of 0.1 mg/l. A January 16, 2001 memorandum (from Ray Anderson of SAIC to Ashley Allen and Gwen DiPietro of EPA, OSW) explains the laboratory reporting limits for TCLP and SPLP thallium analyses and documents our correction of TCLP thallium result for DPE-SO-01. The memorandum is placed in the docket for today's rule.

We then re-examined our analyses of Edge Moor's Iron Rich™ (DPE-SO-01) sample for total and TCLP thallium. Our reported TCLP result (0.28 mg/l) is substantiated by a duplicate analysis (0.27 mg/l) and excellent QC sample results (matrix spike and matrix spike recoveries of 94% and 92%). Our total result (3.7 mg/kg) is probably underestimated, as confirmed by a second analysis (18.4 mg/kg) of the sample which occurred on the same day with 10 times dilution. The analytical batch which contains our diluted and undiluted TCLP thallium results for DPE-SO-1 is placed in the docket for today's rule. DuPont's split total thallium value (23.6 mg/kg) for this sample was even higher than our total thallium results. Table 22-3 summarizes the levels of thallium that we, as well as DuPont, detected in Iron Rich™.

Table 22-3: Summary of Thallium Analytical Results in DuPont Edge Moor Iron Rich™

Edge Moor Iron Rich™	Total (mg/kg)	TCLP (mg/l)
Our sample results	3.7 (18.4[1])	0.28 (0.27[2])
DuPont split sample results	23.6	Not reported

[1] Second analysis occurred on the same day with a 10X dilution

[2] Duplicate analysis

Since two separate portions of the waste sample were used for the analyses of total and TCLP thallium tests, we find that the level of thallium (0.28 mg/l) in the TCLP waste leachate (DPE-SO-01) is actually very close to the maximum theoretical TCLP concentration (i.e., 0.185 mg/l) calculated using our total thallium concentration of 3.7 mg/kg and a dilution factor of 20 (i.e., 3.7/20 = 0.185 mg/l) (see Table 22-4 below). Our thallium TCLP concentration (0.28 mg/l) was much lower than the maximum theoretical TCLP concentration (i.e., 1.18 mg/l), if calculated using our second total thallium concentration of 18.4 mg/kg or DuPont's split total thallium concentration of 23.6 mg/kg.

Table 22-4: Comparison of Theoretical and Actual Thallium Levels in DuPont Iron Rich^{TM} (DPE-SO-01)

Total Thallium (mg/kg)	Thallium Theoretical Maximum Concentration in TCLP Leachate (mg/L)	Actual Thallium Value in Waste TCLP Leachate (mg/L)
3.7	0.185	0.28
18.4 (EPA second analysis)	0.92	
23.6 (DuPont Split)	1.18	not reported

* Thallium theoretical maximum concentration in TCLP leachate = total thallium ÷ 20

DuPont theorized that our TCLP result may be due to contamination based on detection of low level thallium in two continuing calibration samples. While DuPont is correct to point out this low level contamination (3.37 ug/l and 3.2 ug/l), the level of contamination is only slightly above the method detection limit (3.14 ug/l) and still below the reporting limit (5 ug/l). It is lower than our detected thallium TCLP values by a factor of 84. We believe it is unreasonable to qualify a sample result based on trace level contamination slightly above the method detection limit and below the reporting limit. We found no thallium contamination in any other blank samples (preparation blank, method blank, continuing calibration blanks) greater than the method detection limit. Furthermore, our QC sample results demonstrate that our TCLP data has excellent precision and accuracy.

Comment 22-5a-2: EPA's thallium SPLP value for Iron Rich^{TM} is suspect.

In light of the questionable results for thallium TCLP, the very low value of 0.012 mg/L reported by SAIC for thallium SPLP for DPE-SO-01 in Table 4-3 of the SAIC report comes into question.

The thallium SPLP value is not footnoted as being less than the typical laboratory reporting limit. This value of 0.012 mg/L, however, is slightly above the reporting limit of 0.005 mg/L listed in Table 4-3 of the SAIC Report.

As noted above, the review by Environmental Standards of the raw APPL data supplied by EPA indicates blank contamination at a level of 0.0033 mg/L in the CCB for the TCLP analyses. EPA did not provide raw CC13 or preparation blank data for the SPLP analysis.

In light of the apparent external thallium contamination of the TCLP sample, Environmental Standards notes that the reported SPLP thallium value "could also be the result of external contamination." See item 2 of the letter in Attachment 5.1.1-A.

DuPont is interested in obtaining the full APPL report and the remaining raw instrumental data from the SPLP analyses, including the results for all the blanks to assist EPA in further review of the data.

Based on the closeness of the thallium SPLP result to the only blank value available and the artificially high thallium TCLP result, DuPont believes that EPA's thallium SPLP value used in the Agency's risk assessment is a laboratory artifact rather than a real number.

Response 22-5a-2: DuPont argues that EPA's thallium SPLP value for Iron Rich™ is suspect because of the purported TCLP problems. However, DuPont had not seen the entire laboratory raw instrument output for the SPLP analysis. "The raw analytical data for DuPont Edge Moor, Delaware facility" that we placed in the RCRA docket for the inorganic chemical listing proposal on October 25, 2000, contains TCLP and SPLP results for Iron Rich and laboratory QC samples results associated with the TCLP analysis. However, we inadvertently did not include any of the blank or other laboratory QC sample results associated with the SPLP analysis for Iron Rich™.

We re-examined our SPLP thallium results for Iron Rich™ and associated QC samples and found those results were acceptable. Since our TCLP and SPLP tests were run six days apart, DuPont should not cast doubt on our thallium SPLP value for Iron Rich™ based on the purported TCLP problems. We placed our entire laboratory analytical batch results for total, TCLP and SPLP analyses in the RCRA docket for today's notice. Also, as described in response to Comment 22-5a-1, we consider our TCLP thallium results valid, thus this would not be a basis to question to SPLP data in any case.

Comment 22-5a-3: Sampling and analysis of process streams show that thallium is not present in the levels suggested by EPA.

In response to this proposed listing, DuPont has statistically sampled and analyzed designated wastewater and solids streams to determine if there was a basis for including these streams, or the solids that result from them, within the scope of the proposed K178 listing. In all, one hundred and twenty two samples were collected and analyzed for a combination of total, TCLP and SPLP levels of twenty-two metals. A total of two hundred and nine sample analyses and associated QA/QC samples were performed on these samples. The thallium results, summarized in tabular form as Attachment 5.1.3-A, indicate only two quantifiable thallium detections on a total composition basis at slightly above the analytical reporting limit of 0.5 mg/kg. No SPLP results were above the reporting limits of 0.005 - 0.01 mg/L. No TCLP results were above the reporting limits of 0.005 - 0.025 mg/L.

The results from the sampling and analysis program, along with the laboratory QA/QC, is provided in Attachment 5.1.3-B. The sampling plan for the analysis of solids and sludges is presented in Attachment 5.1.3-C. The sampling plan for the analysis of wastewaters associated with wastewater treatment sludges is presented in Attachment 5.1.3-D. The data validation report from Environmental Standards is presented in Attachment 5.1.3-E.

Response 22-5a-3: We appreciate DuPont supplying us with data that characterize Iron Rich^{TM}, wastewater treatment sludges and wastewaters. DuPont collected 61 solids and 44 wastewater samples from 3 facilities, plus QA/QC samples. Samples were analyzed by 2 laboratories. All samples were analyzed using two different analytical procedures (SW 6010B, Inductively Coupled Plasm-Atomic Emission Spectrometry (ICP) and SW 6020, Inductively Coupled Plasma Mass Spectrometry (ICPMS)) for total and SPLP concentrations of 21 metals; 2 streams were also assessed via TCLP. DuPont's analytical data and validation reports (in 11 volumes) and raw laboratory CLP-like data packages (in 9 CDs) are placed in the RCRA docket for today's notice.

We conducted a complete review of DuPont's data to determine the validity of its waste analysis. See "Assessment of Analytical Data Submitted by DuPont In Response to Proposed Inorganic Chemical Industry Hazardous Waste Determination for K178, October 2001" for our determination of the validity of DuPont's waste analysis. The following summarizes our review of DuPont's thallium data for Iron Rich^{TM}, wastewater treatment solids and wastewaters.

First, several major analytical problems were found in DuPont's ICPMS thallium analysis.

> (1) STL's method detection limit (MDL) studies are outdated. STL's laboratory located in West Sacramento, California, which analyzed the Iron Rich^{TM} and wastewater treatment sludge samples, conducted their ICP and ICPMS MDLs and reporting limits (RLs) studies for metals in early 1998[9]. This is unacceptable since the operating conditions, time in service, and maintenance affect the sensitivity of the instrument over time. A more appropriate time frame would have been on a quarterly basis[10]. In addition, the ICPMS Method 6020A used for these analyses requires detection limit studies to be determined at least every three months and kept with the instrument log book[11]. We believe at a minimum the MDL studies should be performed on an annual basis. We were very surprised the STL-West Sacramento, California lab operated and responded to various external audits to

---

[9] STL's laboratory located in Denver, CO, which analyzed all the wastewater samples, conducted their ICP and ICPMS MDLs/RLs studies in early 2000.

[10] Contract Laboratory Program (CLP) labs according to the CLP Inorganics Statement of Work, ILMOS.OC, March 1999, require detection limit studies to be performed on a quarterly basis (January, April, July, and October).

[11] The SW-846 Chapter One guidance does not set a frequency at which MDLs should be updated, however, Section 4.4.1 of Chapter One does state that the laboratory should have procedures in place for demonstrating method proficiency and determining the MDL. Therefore, the laboratory should establish a written policy that meets their certification requirements and allows the laboratory to update QC parameters at a reasonable interval.

establish applicable certifications without a more current MDL study because such audits generally request and review for documentation to support the establishment of proposed detection and reporting limits and verify if the frequency of MDL study updates is appropriate. STL's detection limits for ICPMS and ICP metals should have been adjusted routinely for all associated sample analyses and matrices to reflect changes in target analyte responses over time using the most current MDL data available. Since the MDL study data are outdated, the reported detection limits most likely do not reflect the current instrument operating conditions, sample preparations procedures, and potential sensitivity changes. Therefore, all low-level concentrations (at or near the reporting limit) and non-detect thallium results associated with outdated detection limits should be considered as estimated values and questionable validity.

(2) STL appears not to have used the actual sample weight in their calculations for total ICPMS metals results. Because a method-recommended sample weight (not the actual sample weight) was used in their calculations, the ICPMS results for total metals in all solid samples should only be considered to be accurate to no more than 2 significant figures.

(3) The accuracy of DuPont's “non-detect” and low-level positive thallium in many samples is questionable. Below are the problems we noticed in DuPont's waste analysis.

- ICPMS instrument calibration

STL's ICPMS instruments were calibrated using a single-point calibration, not a calibration curve[12]. The ICPMS instruments (M01 and M02) used to assess Iron Rich^{TM} and pond sludge samples were calibrated using one standard (thallium at 50 ug/l) and optimized at the upper calibration level (thallium at 40 ug/l). The ICPMS instrument (ICPMS1) used to assess wastewater sludge samples was calibrated and optimized using standards at the same level (thallium at 200 ug/l). STL analyzed a practical quantitation limit standard (PQLCRI) for the ICP analysis, however, not for the ICPMS analysis. We could not determine the sensitivity and stability of their ICPMS machines for thallium below their calibration levels (40 ug/l for ICPMS M01 and M02; and 200 ug/l for ICPMS1). With the various analytical problems described in this section that STL had, it seemed very unlikely that STL could have reliably measured thallium at levels in the vicinity of their MDLs (0.1 ug/l, for ICPMS M01 and M02; and 0.02 ug/l, for ICPMS1).

- Blank contamination

Numerous blank samples (e.g., method blanks, initial and continuing calibration blanks) yielding concentrations above (two to five times) the MDL. As a result of this problem, positive results for thallium (as well as other analytes not discussed in this review) of many samples were qualified by ES as “U” (non-detect).

---

[12] SW-846 allows the use of single-point calibration for both ICP (SW 6010) and ICPMS (SW 6020).

- Large negative instrument responses

Many samples had large negative instrument responses for thallium with absolute values greater than three to four times the MDL. ES qualified the "non-detect" sample results with "UJ" indicating that the MDL may have been higher than reported.

- Possible carry-over problems

Many blank samples (method blanks, initial/continuing calibration blanks) exhibited contamination (four to six times the MDL for the thallium analysis associated with the Iron Rich and pond sludge samples). Those blanks were analyzed immediately after an initial or a continuing calibration verification standard (ICV or CCV). There may have been carry-over problems.

Second, we rejected DuPont's ICPMS total, TCLP and SPLP thallium results. We conclude that our data are valid and represent the wastes in question.

DuPont provided analytical data characterizing Iron Rich™ for eight samples (plus 1 duplicate). These samples were comparable to our Iron Rich™ (DPE-SO-01). All 8 samples and the duplicate were analyzed for total, TCLP and SPLP concentrations of 21 metals, including thallium. Table 22-5 shows levels of thallium DuPont reported in their Iron Rich™.

Table 22-5: Thallium in DuPont's Iron Rich™ (ICPMS- SW6020)

Matrix	Total (mg/kg)	TCLP (mg/l)	SPLP (mg/l)
Iron Rich™	<0.18-0.33 U	<0.0005	<0.0005

U This analyte was present in associated blanks. Positive results were qualified as "U" (non-detect).

We believe that all of DuPont's results summarized in Table 22-5 should be rejected. We rejected total thallium results because of the analytical problems described previously. We rejected the TCLP and SPLP data for the same reasons, and we believe these results were further compromised as a result of serious analytical problems (physical and/or memory interferences) that appeared in the ICPMS instrument output on the day that the TCLP and SPLP tests were conducted. Based on our assessment of these output, we believe for both total and TCLP/SPLP tests the lab should have terminated the analyses, corrected the problems, recalibrated the instrument, verified the new calibration, and re-analyzed the affected samples.

DuPont reported two sets of results (ICP and ICPMS) for manganese and reported one set of results (i.e., ICPMS) for thallium. While they did not report ICP results for thallium as part of their formal comments, the analyses were automatically conducted when DuPont ran the ICP analyses for manganese and were submitted as part of DuPont's raw data package. We also reviewed these ICP thallium data to determine whether or not we could make any conclusions from their analyses regarding the presence of thallium in the Iron Rich™. We believe that DuPont's data, generated using ICP, for total and TCLP/SPLP concentrations of thallium may be valid and usable. Table 22-6 compares the EPA and DuPont ICP thallium results.

Table 22-6: Thallium in DuPont's Iron Rich^{TM}, (ICP- SW6010)		
Analysis	EMI-1 through 8	EPA Sample (DPE-SO-01)
Total-6010 B (mg/kg)	<7.1	3.7 (18.4[1] EPA second analysis) (23.6 DuPont split)
TCLP-6010 B (mg/L)	<0.250	0.28 (0.27 EPA duplicate) (DuPont did not report their split result)
SPLP-6010 B (mg/L)	<0.050	0.012 (DuPont did not report their split result)

[1] Second analysis occurred on the same day with a 10X dilution

As Table 22-6 indicates, DuPont found no total or TCLP/SPLP thallium in their Iron Rich^{TM} samples (EMI-1 to EMI-8 and EMI-6-Dup) above the laboratory reporting limits using ICP. We detected total thallium (3.7 mg/kg) in our sample (DPE-SO-01). We detected SPLP thallium in the sample at a level lower than DuPont's corresponding detection limits, and TCLP thallium at a level similar to DuPont's TCLP detection limit.

We believe that the validity of our total thallium value (3.7 kg/mg) is supported by even higher of thallium detected in a second run of our sample (DPE-SO-01) at 10 times dilution of 18.4 mg/kg, as well as by DuPont's split analysis of this sample at still higher level (23.6 mg/kg). (DuPont did not report TCLP and SPLP thallium results for the split sample.) Therefore, we conclude that our data are valid and representative of the waste in question.

Third, we suspect the validity of DuPont's analytical results for thallium and we do not agree with DuPont's assertion that thallium is not present in the wastewater treatment solids. The detected thallium leachate values, however, are below the MDL and therefore, would not be used to support a regulatory determination.

DuPont collected 44 wastewater samples to characterize wastewaters (11 streams) and 53 pond sludge samples to characterize their wastewater treatment sludges (6 streams) resulted from the treatment of wastewaters. Their pond sludge samples were comparable to our sample (DPN-SO-01) of wastewater treatment sludge collected from Johnsonville's Hillside Pond. Tables 22-7 and 22-8 show levels of thallium DuPont reported in their wastewater treatment solids and wastewaters.

Table 22-7: Thallium in DuPont's Wastewater Treatment Solids (ICPMS- 6020)			
Matrix/Analysis	Total (mg/kg)	TCLP (mg/l)	SPLP (mg/l)
Edge Moor Wastewater Treatment Sludge	<0.9-0.22	NA	<0.0005-<0.001

Table 22-7: Thallium in DuPont's Wastewater Treatment Solids (ICPMS- 6020)

Matrix/Analysis	Total (mg/kg)	TCLP (mg/l)	SPLP (mg/l)
Johnsonville Hillside Pond	<0.018-0.43	<0.0005-0.0017	<0.0001-0.00098
Johnsonville Equalization Pond	<0.018	NA	<0.0001-0.00045
Johnsonville Settling Pond	<0.18	NA	<0.0001
DeLisle Equalization Basin	<0.18-0.84	NA	<0.0001
DeLisle Disengagement Basin	<0.18-0.59	NA	<0.0001

Table 22-8: Thallium in DuPont's Wastewaters (ICPMS- SW6020)

Matrix/Analysis	Total (mg/l)
Johnsonville Wastewaters	<0.0002-<0.002
DeLisle Wastewaters	<0.0002-<0.002
Edge Moor Wastewaters	<0.00002-0.00057

DuPont's analytical results for the wastewater treatments solids and wastewaters generated at all three facilities indicate that no wastewater treatment sludge samples contain leachable thallium at levels above the HBL (0.001 mg/l) nor do any wastewater samples contain total thallium at levels above the HBL.

As described earlier, there were analytical problems (i.e., instrument calibration, blank contamination, large negative instrument responses, possible carry-over problems) commonly found in DuPont's ICPMS thallium analysis. Therefore, we had no way to determine the accuracy of all DuPont's non-detect and low ICPMS thallium results for wastewater treatment solids and wastewaters.

We believe, however, that DuPont's ICP (SW 6010) thallium data for wastewater treatment solids and wastewaters may be valid. Table 22-9 compares DuPont and EPA ICP thallium results for wastewater treatment solids. Table 22-10 shows levels of total thallium in DuPont's wastewaters, as measured by ICP analysis.

Table 22-9: Thallium in DuPont's Wastewater Treatment Solids (ICP- SW 6010)

Matrix/Analysis	Total (mg/kg)	TCLP (mg/l)	SPLP (mg/l)
Edge Moor Wastewater Treatment Sludge	<35.5	NA	<0.053 (2 samples) 0.0019-0.033* (6 samples)
Johnsonville Hillside Pond	<7.1-<14.2	<0.27 (2 samples) 0.01-0.16* (6 samples)	0.0015-0.041*

Table 22-9: Thallium in DuPont’s Wastewater Treatment Solids (ICP- SW 6010)

Matrix/Analysis	Total (mg/kg)	TCLP (mg/l)	SPLP (mg/l)
Johnsonville Equalization Pond	<7.1	NA	<0.053
Johnsonville Settling Pond	<7.1	NA	<0.053
DeLisle Equalization Basin	<7.1	NA	<0.053
DeLisle Disengagement Basin	<7.1	NA	<0.053
EPA sample (DPN-SO-01)	7.2	<0.1	<0.0022/<0.01 (split)

Table 22-10: Thallium in DuPont’s Wastewaters (ICP- SW6010)

Matrix/Analysis	Total (mg/l)
Johnsonville Wastewaters	<0.0049
DeLisle Wastewaters	<0.01 (7 samples) <0.1 (1 sample) 0.0047-0.0275J (9 samples)
Edge Moor Wastewaters	<0.0049

* The result is greater than instrument detection limit (IDL) but less than the method detection limit (MDL).
J This result is greater than the IDL and/or MDL but less than the laboratory reporting limit (RL).
NA Not analyzed.

Table 22-9 shows that no total thallium was detected in DuPont’s wastewater treatment solids above the laboratory SW6010 MDLs. It also indicates that TCLP thallium was detected in DuPont’s Johnsonville Hillside Pond sludge and SPLP thallium was detected in DuPont’s Edge Moor wastewater treatment sludge and Johnsonville Hillside Pond sludge; each of these detections, however, were below the MDL of 0.053 mg/L. We detected total thallium in our sample (DPN-SO-01) at a level close to DuPont’s corresponding detection limit; we did not detect TCLP thallium at a level lower than DuPont’s TCLP detection limit.

Table 22-10 shows that low levels of total thallium were detected in DuPont’s DeLisle wastewaters. These levels are unlikely to contribute significantly to the thallium concentrations of sludge.

In summary, we have significant concerns about DuPont’s ICPMS thallium analysis. We suspect the validity of DuPont’s ICPMS results for thallium. DuPont’s ICP total, TCLP and SPLP thallium results conform to our ICP total, TCLP and SPLP thallium results. We do not agree with DuPont’s assertion that thallium is not present in the wastewater treatment solids. The detected thallium leachate values, however, are below the MDL and therefore, would not be used to support a regulatory determination.

<u>Comment 22-5a-4:</u> Process knowledge confirms that thallium is not expected in process

solids at the levels suggested by EPA for any chloride-ilmenite facility.

DuPont analyses of the ores used in the manufacturing process for various ores used in the past year indicate that thallium is generally not present at levels above 50 ug/kg. The highest level we have identified in any ore is 171 ug/kg, which would equate based on solids/ore ratio to a theoretical maximum of 350 ug/kg on a total composition basis for any solid created from the process. This correlates to the data results on thallium summarized above and presented in Attachment 5.1.3-A.

Response 22-5a-4: DuPont's analysis is not convincing because: (1) DuPont did not present their ore analyses for our review; (2) they limited their review to one year; and (3) DuPont did not assess their other primary raw material, petroleum coke, for thallium.

Data from prior analyses by EPA and submitted by DuPont confirms that thallium has been present in the Iron Rich™ and similar wastes at levels significantly above levels they estimate from their ore analyses:

Sampling event	Thallium results (total)
DuPont analysis of the split from EPA's 1999 sample of Iron Rich™	23.6 mg/kg, wet weight; 58.7 mg/kg, dry weight
Geosystems sample of stabilized Iron Rich™, 5/12/99	28.6 mg/kg
EPA sample of New Johnsonville WWT solids (DPN-SO-01)	7.2 mg/kg
EPA sample of Edge Moor ferric chloride (DPE-SO-01)	3.7 mg/kg

In summary, we disagree with DuPont's assertion that thallium is not present in their wastes. We continue to believe that thallium is present in the Iron Rich™ and that thallium is an appropriate basis for listing nonexempt ferric chloride solids.

Comment 22-5b: EPA should revise the K178 listing regulatory language to make it consistent with the preamble in the proposal.

The Agency has proposed that the listing description for K178 should be defined as "Nonwastewaters from the production of titanium dioxide by the chloride-ilmenite process. (This listing does not apply to chloride process waste solids from titanium tetrachloride production exempt under 40 CFR 261.4(b)(7).) (T)."

That listing description would have the effect of listing, as hazardous wastes, several waste streams that the Agency has explicitly excluded in the preamble language. Specifically, solids from the finishing area wastewaters are included in the language as defined in the proposal, but off-specification Ti02 product (which is what the solids in the finishing wastewaters are) was determined not to be a waste eligible for listing in the preamble language contained at 65 FR 55765. While preamble language is helpful to the regulated community in determining the Agency's intent when determining regulatory compliance strategies, the regulatory language is the basis for Agency enforcement of the regulations.

DuPont requests that EPA revise the listing to capture only those wastes for which the proposal has included data indicating that the waste poses a hazard to human health and the environment.

Response 22-5b: The regulatory language in the final rule accurately defines the waste intended to be listed as hazardous waste K178 and is consistent with the explanation of the hazardous waste listing provided in the preamble to the final rule. We note that the listing description as finalized is different than the proposed listing description referred to by the commenter and the potential concern raised by the commenter is now moot.

Comment 22-5c: DuPont understands that the term "wastewater treatment solids" means solids that are separated from wastewater via a wastewater treatment process rather than solids contained in the wastewaters prior to treatment.

The rationale for not listing commingled wastewaters from the chloride-ilmenite process is as presented at 65 FR 55763 through 65 FR 55765 with the conclusion that these commingled wastewaters do not merit listing. DuPont agrees that these wastewaters do not merit listing and requests that the Agency clarify that no listing applies to these wastewaters as a result of the fact that they contain, prior to wastewater treatment, solids which the Agency has proposed for listing, if such a listing is finalized. Absent such clarification, wastewaters evaluated by the Agency and deemed to not warrant listing may be inadvertently captured as listed wastes by virtue of contact with listed wastes.

Response 22-5c: In the final rule, EPA did not list wastewaters from the production of titanium dioxide, or any other wastewaters, as hazardous waste. Therefore, the commenter is correct that the commingled wastewaters from the chloride-ilmenite process are not listed hazardous wastes. However, we clarify that any wastewaters that are mixed with, or managed in a manner that involves mixing with, any other listed hazardous waste, including the solids from chemical manufacturing of ferric chloride that are listed in today's final rule, are hazardous wastes by virtue of the hazardous waste "mixture rule" (40 CFR 261.3(a)(2)(iv)).

Comment 22-5d: Wastewater treatment solids should be excluded from the listing.

The Agency has proposed to include wastewater treatment solids in the scope of the listing. The listing is based on a misinterpretation of the submitted §3007 information that these sludges are synonymous with the metal chloride condenser solids and that they contain significant levels of the metals of interest (manganese and thallium) which form the basis of the listing. Those conclusions are incorrect for the following reasons.

Comment 22-5d-1: The Agency's assumption that the Delaware facility's wastewater treatment solids are the same as Iron Rich™ is inaccurate.

The Agency indicates at 65 FR 55761 that the data in Table III-54 characterizes wastewater treatment solids at the Delaware facility, yet this data is for DPE-SO-01, which is a sample "directly from the Iron Rich™ dewatering press". Iron Rich™ contains only 10% wastewater treatment solids at the Delaware facility, but the analytical data for Iron Rich™ is not characteristic of wastewater treatment solids from the Delaware facility. Iron Rich™ is primarily composed of coke and ore solids. DuPont asserts that the coke and ore solids and the wastewater treatment are not chemically similar, and this assertion is confirmed by the analysis of wastewater treatment solids at the Delaware facility.

The data DuPont has collected in response to this proposed rulemaking included analysis of both the Iron Rich™ product and the wastewater sludge prior to combination of these materials. The comparison of this data yields the following data.

Table 5.1

Comparison of Delaware Wastewater Sludge and Iron Rich™
Total Composition Basis

Constituent Concentration, mg/kg	Wastewater Sludge	Iron Rich™
Manganese	30	14,000
Thallium	ND	ND
Iron	660	130,000
Vanadium	43	250

Table 5.2

Comparison of Delaware Wastewater Sludge and Iron Rich SPLP Basis

Constituent Concentration, mg/1	Wastewater Sludge	Iron Rich
Manganese	0.1	12.6
Thallium	ND	ND
Iron	0.032	.48
Vanadium	ND	ND

This data clearly indicates that the wastewater sludge is compositionally a fundamentally different material than the Iron Rich™ product and that it further does not warrant listing based on the absence of appreciable metals of concern on a total composition basis and the relatively low leachability of metals in the sludge. To list these solids, which clearly pose no hazard based on metals analysis, creates a disincentive to separate management of these solids if the facility should determine that separate management of the wastewater sludge is appropriate. To assert, as the Agency does at 65 FR 55763, that these wastewater sludges, if separately managed from the Bevill exempt solids included in the Iron Rich^{TM} product, would pose risks is without basis. The wastewater solids were not sampled as part of the Agency information collection and therefore, no basis to assess the risk posed by them was available to the Agency at time of proposal. The above data clearly indicates that no appreciable manganese nor thallium is contained in the wastewater treatment sludge. Specifically, comparison of the SPLP result to health based levels for thallium and manganese would have screened out if evaluated under the same procedures used to develop Table 111-54 presented at 65 FR 55761. Therefore, the risk assessments presented at 65 FR 55762 and 65 FR 55763 and labeled Tables 111-56 and 111-57 are not driven by, nor are equitable to, wastewater treatment solids from the Delaware site.

The Agency should include in the listing only those streams for which the risks are shown to be unacceptable. The Agency has not sufficiently demonstrated an unacceptable risk for wastewater treatment solids at the Delaware facility. The solids which are generated from the chloride-ilmenite wastewater treatment systems are generally from caustic and water scrubber purges which contain little or no metals (based on vendor supplied analysis of the caustic detailed below) and are therefore not sources of the metals of concern to the Agency based on the proposal. The only process related solids in these streams, as the Agency noted at 65 FR 55751, are Bevill exempt carryover solids. Since the Agency has acknowledged that Bevill exempt solids are not candidate for listing in this proposal, there is no basis on which these wastewater sludges are candidate for listing.

<u>Response 22-5d-1:</u> After reviewing comments and data submitted by DuPont in response to the proposed rule, we evaluated all available data on each component waste included in the combined Iron Rich^{TM} wastestream against our hazardous waste listing criteria. In making our final listing determination, the wastewater treatment solids component of the wastestream was evaluated separately from the other components of the combined wastestream. We thank the commenter for its submission of sampling and analytical data, which facilitated this evaluation.

We are finalizing a decision *not* to list the non-exempt wastewater treatment solids. We note that, although manganese was a constituent of concern in our proposal, we are deferring action on manganese for the final rule. See Section IV.B. of the preamble. We also note that, if manganese had remained a constituent of concern, and if we had used the same RfD and HBL that we used in the proposal, we would have concluded that manganese in these solids did not pose significant risks. For the remaining constituents, as explained in the preamble to the final rule, after reviewing all the analytical data submitted by DuPont, we have concluded that the non-exempt portion of the wastewater treatment solids generated from the production of titanium dioxide does not contain manganese at levels of concern.

! DuPont submitted data supporting a conclusion that we should have used a higher K_d value for thallium in our risk analysis for this wastestream. Given that the higher K_d value would result in a larger DAF and a lower potential risk from thallium, we further conclude that the non-exempt portion of the wastewater treatment solids poses no significant risk due to the presence of thallium.

- As discussed in the preamble, we assessed DuPont's data that indicated that antimony may be present in these wastes. We constructed a worst case screening scenario that predicts that the non-exempt portion of these wastes would not create significant risk:
  - Initially, we calculated a maximum theoretical TCLP value using the maximum total antimony value reported for the wastewater treatment sludge (i.e., 3.8 mg/L divided by 20), yielding a worst case TCLP value of 0.19 mg/L. While this value clearly exceeds the antimony HBL of 0.006 mg/L, we recognized that the wastewater treatment sludge is comprised of exempt and non-exempt components, and that some proportion of this HBL exceedance would be associated with the exempt solids that are outside the scope of this listing determination.
  - To isolate the portion of the risk that is associated with the non-exempt wastewater treatment sludges derived from treatment of oxidation and finishing wastewaters, we used DuPont's antimony analytical data for its major oxidation and finishing wastewater (RIN 13, dryer scrubber water)[13] to estimate what the concentration of antimony would be in the wastewater treatment sludge if (1) all of the antimony in this wastewater were concentrated in the sludge, and (2) this wastewater was the only source of antimony contributing to the sludge antimony concentration. We estimated this maximum theoretical total concentration of antimony from oxidation and finishing wastewaters in the wastewater treatment sludge to be 0.036 mg/kg in the following manner:

    $3.8\ \text{ug/L}_{\text{average antimony concentration in RIN 13, ICP analysis}}\ \times\ 124{,}259\ \text{MT/yr}_{\text{RIN 13 wastewater volume}}$
    $\times\ (\text{L/kg}\ \times\ 10^3\ \text{kg/MT}\ \times\ \text{g}/10^9\ \text{ug}\ \times\ \text{kg}/10^3\ \text{g}\ \times\ \text{MT}/10^3\ \text{kg})_{\text{unit conversions}}$
    = 0.00047 MT antimony in RIN 13. If all of the antimony loading in RIN 13 were concentrated in the sludge, the antimony concentration in the sludge due to RIN 13 would be:

    $0.00047\ \text{MT antimony} / 13{,}000\ \text{MT wastewater treatment solids} = 0.036\ \text{ppm antimony}_{\text{maximum theoretical total antimony concentration in wastewater treatment sludge}}$
  - This concentration is significantly lower than the measured antimony levels in the total wastewater treatment sludge samples, indicating that the non-exempt portion of the wastewater treatment sludge does not contribute much antimony loading to the overall sludge volume.
  - Finally, to complete this screening analysis, we projected a maximum theoretical TCLP value of 0.002 mg/L from the maximum non-exempt antimony sludge concentration by dividing the total value by 20. This TCLP maximum value is below the HBL of 0.006 mg/L.
  - We conclude from this analysis that it is unlikely that the non-exempt portion of the wastewater treatment sludge would pose risk from antimony if the waste were placed in a municipal solid waste landfill.

---

[13] See Appendix C of *Assessment of Analytical Data Submitted by DuPont in Response to Proposed Inorganic Chemical Industry Hazardous Waste Determination for K178*, October 2001.

- Also, although data submitted by DuPont indicates that arsenic may be present in the wastewater treatment solids generated at the Mississippi facility at levels of concern, we are uncertain regarding the source of the arsenic in the combined wastewater treatment solids. In addition, due to the fact that the DAF for arsenic at the Mississippi site is relatively high (as calculated for the wastewaters in support of the proposal), we believe that potential risks due to the presence of arsenic are below levels of concern. The Agency therefore is finalizing a no list determination for the non-exempt portion of the wastewater treatment solids generated by the chloride-ilmenite process.

Comment 22-5d-2: The Agency has erroneously used collected solids data from the Delaware and Tennessee sites by applying it to the Mississippi site

DuPont disagrees with 65 FR 55760 of the proposed rule, the Agency states that the sampling and modeling of the Tennessee and Delaware sites are appropriate surrogates for the Mississippi facility, given the similar nature of the processes at the three facilities (with particular asserted similarities between the wastewater treatment facilities at Mississippi and Tennessee).

The Tennessee facility indicates in the RCRA §3007 Survey response that purification area scrubber water (RIN 3), reaction area maintenance scrubber (RIN 103), recovered ore scrubber wastewater (RIN 4), reaction start-up scrubber wastewater (RIN 102), and reaction maintenance wastewater (RIN 109) are managed in the wastewater treatment system. The Mississippi Facility reports in the RCRA §3007 Survey response that streams similar in nature to these Tennessee facility streams are collected in the recycle water storage system. The recycle water storage system segregates these streams from the Mississippi facility's wastewater treatment system. The only streams from the reaction process that enter the wastewater treatment system at the Mississippi facility are the chlorine unloading scrubber wastewater (RIN 10 1), emergency chlorine scrubber wastewater (RIN 102) and pretreated reaction scrubber water from the final reaction area scrubbing stage (RIN 3). Therefore, the resulting wastewater treatment sludges are generated from different wastewater streams at the two facilities.

In summary, the most significant difference in the wastewater treatment facilities is the management of wastewater containing coke and ore solids from the reaction processes. At the Tennessee facility, wastewaters containing Bevill-exempt coke and ore solids are commingled with oxidation and finishing wastewater. The same Bevill-exempt solids are managed in the recycle water and environmental filtration systems at the Mississippi facility, and these solids are not commingled with solids from the wastewater treatment system.

The data comparison of wastewater treatment sludges below confirms the differences in the three facilities.

Table 5.3 Wastewater Treatment Sludges

Constituent Concentration, mg/kg	DuPont Site		
	Delaware	Mississippi	Tennessee*
Manganese	30	3	2,890
Thallium	ND	ND	7.2**
Iron	660	80	63,200
Vanadium	43	20	1,060

* From 65 FR 55761, Table 3.27 in the Background Document

** Shown in Attachment 5.1.1-A to be the result of a laboratory artifact

The high levels of metals in the Tennessee sample are due to the presence of materials EPA determined to be outside of the scope of this listing determination.

EPA should use site-specific data to model and to make listing determinations for wastewater treatment sludge at the Mississippi Facility. The sampling and analysis plan used to generate the data is provided in Attachment 5.1.3-C.

<u>Response 22-5d-2:</u> As explained in the preamble to the final rule, we are promulgating a hazardous waste listing only for the ferric chloride solids. In the final rule, we are not listing wastewater treatment solids or condenser solids generated by the chloride-ilmenite process as hazardous wastes. Therefore, the only plant that generates the listed hazardous waste is DuPont's Delaware facility. The waste generation and management practices for wastewater treatment solids at the Tennessee and Mississippi plants, as well as the site-specific characteristics of the these two facility locations, are not directly relevant to the potential risks from the ferric chloride solids. We note that, although manganese was a constituent of concern in our proposal, we are deferring action on manganese for the final rule. See Section IV.B. of the preamble.

<u>Comment 22-5d-3:</u> The Tennessee facility wastewater treatment solids should not be included in the listing.

At 65 FR 55761, the Agency presents data for wastewater treatment solids at the Tennessee facility. At 65 FR 55762 the results of the screening level analysis for groundwater to surface water risk assessment demonstrate that "concentrations of the constituents of concern in the river are likely to be well below the national AWQC for human health and aquatic life for these constituents." The Agency based the listing decision for the Tennessee facility's wastewater treatment solids on the risk assessment results for Iron Rich^{TM} at the Delaware facility. DuPont contends that this comparison is inappropriate. As stated above, the Agency should include in the listing only those streams for which the risks are shown to be unacceptable. The Agency has determined in their own analysis that the Tennessee facility's

wastewater treatment solids posed no risk yet still identified them for listing by transfer of data from another facility.

Response 22-5d-3: See our response to comment 22-5d-2.

Comment 22-5e: The EPA has listed nonwastewaters from the following chloride ilmenite process streams without basis

Because this listing includes solids generated from nonspecific sources (contrary to previous Agency use of the K-listed waste criteria for specific wastes), any solids generated out of the following waste streams from the chloride ilmenite process would fall within the listing, even though these streams were not sampled by the Agency and the preamble discussion indicates that the Agency intended not to list these solids. These streams were sampled and analyzed by DuPont in response to this proposed rulemaking for twenty-two metals. The results of the analyses for Mn and Tl are presented with each stream below, along with the annual generation rates for each stream as reported by DuPont in its 3007 submissions. The data presented below indicates that none of these wastewater streams are a source of either of the two metals that are the basis of this listing. Therefore, solids from any of these waste streams should clearly not be part of the listing. The remainder of the analytical results is presented in Attachment 5.1.3-B.

Delaware facility

- Oxidation vent scrubber wastewater (RIN 9)
  - Annual stream generation rate (Mtons/year) -59,589
  - Suspended solids - 82 mg/L
  - Manganese (ug/L) - 1.12
  - Thallium (ug/L) - ND

- Finishing Wastewater
  - Annual stream generation rate (Mtons/year) - 4.53
  - Suspended solids - 442 mg/L
  - Manganese (ug/L) - 0.744
  - Thallium (ug/L) - ND

Tennessee facility

- Oxidation scrubber wastewater (RIN 104)
  - Annual stream generation rate (Mtons/year) - 6
  - Suspended solids - 3000 mg/L
  - Manganese (ug/L) - 26.7
  - Thallium (ug/L) - ND

Finishing Wastewater

    Annual stream generation rate (Mtons/year) - 6566998
    Suspended solids - 50 mg/L
    Manganese (ug/L) - 2.6
    Thallium (ug/L) - ND

Mississippi facility

Oxidation scrubber wastewater (RIN 6)

    Annual stream generation rate (Mtons/year) - 9259
    Suspended solids - 41 mg/L
    Manganese (ug/L) - 40.26
    Thallium (ug/L) - ND

Finishing Wastewater

    Annual stream generation rate (Mtons/year) - 2859842
    Suspended solids - 25 mg/L
    Manganese (ug/L) - 17.2
    Thallium (ug/L) - 0.035

<u>Response 22-5e:</u> As a result of the revisions to narrow the proposed scope of the K178 listing, the commenter's concerns are no longer relevant. The final K178 listing focuses only on solids from the production of ferric chloride and is in no way linked to the generation of solids from wastewaters associated with titanium dioxide production. We note that, although manganese was a constituent of concern in our proposal, we are deferring action on manganese for the final rule. See Section IV.B. of the preamble. We also note that, if manganese had remained a constituent of concern, and if we had used the same RfD and HBL that we used in the proposal, we would have concluded that manganese in these solids did not pose significant risks.

<u>Comment 22-5f:</u> Chlorine Scrubber Wastewater and Wastewater Solids

DuPont had originally intended to sample the chlorine scrubbers as well to confirm lack of appreciable metals in those wastes that are also handled in the site wastewater treatment systems. However, due to the concentrated caustic nature of these streams and the anticipated analytical difficulties, DuPont contacted the vendors who supply the sites with caustic and obtained metals analyses from those companies. Those analyses are presented below:

The only appreciable metals expected to be present in caustic are nickel and iron from corrosion in the evaporators used to concentrate the product.

Below are the typical caustic analyses by DuPont routine suppliers:

Table 5.4 Vendor Caustic Analysis, Average (ppm)

Metal	Vendor 1	Vendor 2	Vendor 3
As	0.018	<1	<0.2
Cr	0.087	0.28	<0.5
Mn	0.05	0.06	<0.1
V	0.092		

Note: Caustic vendors do not analyze Thallium

[See original comment for attachments to comment 22-5.]

Response 22-5f: EPA thanks DuPont for this additional clarifying information and notes that it is not relevant given the narrowed scope of the final rule. We note that, although manganese was a constituent of concern in our proposal, we are deferring action on manganese for the final rule. See Section IV.B. of the preamble.

Comment 22-6: Manganese is an essential micronutrient and should not be designated as an Appendix VIII hazardous constituent.

EPA is proposing (65 FR 55687, September 14, 2000) to add manganese to the list of hazardous constituents in Appendix VIII to 40 CFR 261.

Regarding criteria for addition to Appendix VIII, EPA at existing 40 CFR 261.11 states, "Substances will be listed on Appendix VIII only if they have been shown in scientific studies to have toxic, carcinogenic, mutagenic, or teratogenic effects on humans or other life forms."

EPA's language for determining what should be listed on Appendix VIII is too broad to include or exclude any substance on the basis of its "toxicity", since anything (including water) is_ toxic, if you ingest enough of it. The old addage is true, "The dose makes the poison."

DuPont contends that manganese is an essential micronutrient well regulated by the human body and that manganese does not meet the definition of "toxicity" necessary to be classified as an Appendix VIII substance.

Response 22-6: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-6a: Manganese is an essential micronutrient.

According to the 1999 edition of the most widely used basic nutrition college text in the United States, *Understanding Nutrition* by Whitney and Rolfes:

> The human body contains a tiny 20 milligrams of manganese, mostly in bones and metabolically active organs such as the liver, kidneys, and pancreas. Manganese acts as a cofactor for many enzymes that facilitates dozens of different metabolic processes. For example, manganese metalloenzymes assist in urea synthesis, the conversion of pyruvate to a TCA cycle compound, and the prevention of lipid peroxidation by free radicals.

The Food and Nutrition Board of the National Research Council *(Recommended Daily Allowances, 10th* edition, 1989) has established an estimated safe and adequate daily dietary intake for manganese to foster adequate dietary consumption of this essential trace element. The section on manganese in this RDA Handbook opens with the following statement

> Manganese has been shown to be an essential element in every animal species studied.

Indeed, manganese is an essential element is all biological systems, and adverse biological effects occur in both plants and animals, including humans, when the amount of manganese is less than the minimum daily requirement. It is an essential component of the daily human diet, necessary for such functions as the formation of connective tissue and bone, the metabolism of carbohydrates (sugars and starches) and lipids (fats), and neurological development and function. Because it is an essential element, human tolerance for widely varying exposure is much more robust. The biochemical systems that control manganese levels in the body, specifically when exposure occurs via ingestion, are capable of mediating large amounts for long periods of time (Valasquez et al, 1989).

Few, if any, cases of manganese over-exposure in humans have been reported. However, it is well known that deficiencies in essential chemicals at critical times of development nearly always result in lifelong deficits. Moreover, such effects are often insidious, occurring slowly, and unnoticeably over time, until the deficit is so large that it becomes clinically or biologically significant. Thus, EPA's current characterization of its concern for release of manganese to the environment, which could promote public aversion to manganese "exposure," must be revised.

Response 22-6a: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today’s final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-6b: EPA has not demonstrated via scientific studies that manganese has toxic, carcinogenic, mutagenic, or teratogenic effects on humans when ingested in drinking water and therefore has not shown that manganese warrants addition to Appendix VIII.

EPA has compiled an extensive health assessment in support of inhalation hazard in the 1996 IRIS substance file for manganese used as the basis for the addition to Appendix VIII in the proposed rulemaking. The inhalation hazard described by EPA and ATSDR is already addressed via the inclusion of manganese as a Hazardous Air Pollutant under the Clean Air Act. However, these data do not support a designation of hazardous based on oral toxicity. In fact, neither the IRIS substance profile nor the ATSDR draft *Toxicological Profile for Manganese* (September 1997) establishes a conclusive link between manganese in drinking water ingestion and toxicity to humans.

The IRIS substance file reviewed the available data on manganese toxicity and determined the critical effect to be on the CNS system. However, the document concluded that manganese is a ubiquitous element, essential for normal physiological functioning and developed a reference dose (RfD) based on what is considered a safe level as opposed to toxicity. This safe level or no observed adverse effect level (NOAEL) was determined based on the following results:

The National Research Council (1989) determined an "estimated safe and adequate daily dietary intake" of 2 - 5 mg/day. Others (Freeland-Graves, 1987) considered these as too low and recommended 3.5 - 7 mg/day. A survey of diets by WHO (1973) concludes that 8 - 9 mg/day for adults is "perfectly safe".

With a safe level of 10 mg/day and the recognition in the IRIS file that some individuals may, in fact, consume a diet that contributes more that this without any harm, supports the premise that Mn is not "toxic" and should not be listed for this reason. Thus, DuPont believes that the Agency's decision to list Mn is not supported as evidenced below.

The IRIS substance file (page 6 of 37) refers to a 1941 report by Kawamura et al *(Kitasato Arch. Exp. Med* 18: 145-169) as "the only epidemiologic study describing toxicologic responses in humans consuming large amounts of manganese dissolved in drinking water." Velazquez and Du (in *Risk Assessment of Essential Elements,* Mertz, 1994) review the Kawamura paper and note that the "concentration of manganese at the time of exposure was probably at least 28 mg Mn/L" and further that "the total intake was at least 58 mg Mn/day". These levels are as much as an order of magnitude higher than the ESADDI. Further, referring to the study by Kawamura, ATSDR concludes that, "Thus, while there is no doubt these people were exposed to manganese, there is considerable doubt that all of the features of this outbreak (particularly the deaths) were due to manganese alone." (p. 45)

The IRIS substance file also discusses neurological effects suggested to be related to chronic intake of drinking water containing 1.8 to 2.3 mg/L of manganese in northwest Greece (p. 4 of 37). However, the IRIS file concludes that the limitation of the study precludes its use to determine a quantitative dose-response relationship. Instead, the study only provides anecdotal evidence that manganese at these levels causes neurological symptoms. This study was performed with only older adults (50+ years) who also had manganese in the diet of at least 5-6 mg/day (and possibly more) based on their high consumption of vegetables. DuPont contends that taking into account that the Rouff (1995) evaluation showed no significant between water and food ingestion, the combined intake of 7 - 8 mg/day is within the norm of expected intake. The neurological symptoms observed are not specific to manganese and, indeed, are typical of other symptoms of aging. The differences noted with neurological symptoms in an older population are likely to have other root causes.

Further, a study by Vieregge et al (1995) directly contradicts the findings by Kondakis. This study found no differences on neurologic impacts of chronic oral intake of manganese in well water between subjects exposed to maximum manganese concentrations in well water ranging from 0.3 -2.16 mg/L and those in the control group, exposed to manganese concentrations of 0.05 mg/L.

ATSDR (p. 47) reviews the same study by Kondakis et al *(Arch. Environ. Health,* 1989) and concludes, "Thus, this study supports but does not prove that chronic oral intake of manganese can lead to neurological changes in humans."

One possible reason for the apparent lack of manganese toxicity to humans via oral ingestion is that iron and calcium in the diet or in dietary supplements limit manganese absorption (Whitney and Rolfes, 1999). Greger *(NeuroToxicology,* 1999) elaborates, "... the body is protected against manganese toxicity by low absorption and/or rapid presystemic elimination of manganese by the liver but not the kidneys."

The International Programme on Chemical Safety of the World Health Organization *(Concise International Chemical Assessment document on Manganese and its Compounds,* 1999) sums up the overall situation stating, "The available evidence for adverse effects associated with chronic ingestion of excess manganese is suggestive but inconclusive."

Response 22-6b: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-7: While DuPont Concurs with EPA's Risk Threshold And Exclusion of Virtually All Hazardous Constituents From The Listing Basis in The Proposal, Evaluation of The Risk Assessment Underlying The Proposal Indicates That The Risk Assessment Is Flawed And Does Not Support The Proposed Listing.

Comment 22-7a: DuPont concurs with and supports the Agency's use of a HQ of 1 for the 90th percentile of the exposure distribution as the risk threshold basis used in this rulemaking.

Comment 22-7a-1: EPA appropriately used a noncancer HQ of 1 as the level of concern for this listing.

In explaining EPA's listing rationale for non-exempt portions of wastewater treatment solids generated at chloride-ilmenite facilities at 65 FR 55762, EPA indicates that the Agency is using exceedance of a hazard quotient (HQ) of 1 at designated percentile as the specific basis for listing.

In Section 6.3.4 of the Risk Assessment Background Document, EPA reiterates that the level of concern for EPA hazardous waste listings with respect to noncarcinogens is an excess lifetime cancer risk estimate equal to a noncancer HQ of 1.

As EPA stated at 65 FR 55695, "Usually, doses less than the RfD ($HQ < 1$) are not likely to be associated with adverse health risks and therefore, are less likely to be of regulatory concern." Additionally, in some cases, such as where the uncertainty factor used with the RfD (reference dose) is very high due to interspecies extrapolation, the likelihood of adverse health risks may be acceptably low even when the HQ is greater than 1.

A noncancer HQ of 1 has been used as the level of concern in other recent hazardous waste listings.

DuPont supports EPA's use of a noncancer HQ of 1 as the level of concern in this rulemaking.

Response 22-7a-1: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule. EPA, however, is responding to ths comment because it raises issue that may be relevant to other constituents of concern DuPont's wastes. EPA acknowledges the commenter's support of the use of a hazard quotient of 1 as a level of concern. This level of concern corresponds to the Reference Dose (RfD) for the compound of interest. As explained elsewhere, the RfD is an estimate (with uncertainty spanning perhaps an order of magnitude) of a daily exposure to the human population (including sensitive subgroups) that is likely to be without an appreciable risk of deleterious effects during a lifetime. The RfD is useful as a reference point from which to gauge the potential effects of a chemical compound at other doses. Usually, doses less than the RfD are not likely to be associated with a risk of adverse health effects. As the frequency and/or magnitude of the exposures exceeding the RfD increase, however, the probability of adverse effects in the population increases. For this reason, exposures above the RfD are more likely to be of regulatory concern.

Comment 22-7a-2: EPA appropriately evaluated HQ for the 90th percentile of the exposure distribution but has confused the issue by co-presenting the 95th percentile values.

EPA reports HQ results for certain metals as determined from groundwater pathway risk assessment for "non-wastewaters from chloride-ilmenite process" at both the 95th percentile and the 90th percentile (65 FR 55762).

In its discussion of how the groundwater pathway was evaluated for the listing proposal, the Agency states, "EPA defines high end as the 90th percentile and greater of the distribution of exposures in the population." (65 FR 55697)

In the 1990 Toxicity Characteristic rulemaking, EPA used the 85th percentile as the basis for rulemaking. The probabilistic analysis used in the risk assessment for the K178 proposal models high end individual parameters with 90th percentile values. As noted in the Chlorinated Aliphatics final rule (65 FR 67073, November 8, 2000), EPA's Guidance for Risk Characterization (EPA Science Policy Council, 1995) supports the use of the 90th percentile of the exposure distribution for the high end exposure.

Therefore, the Agency has a sound basis for using a threshold of HQ of 1 for the 90th percentile of the exposure or risk distribution in the final listing determination for this rulemaking. And it would be helpful for the Agency to clarify that any HQ results presented at other percentiles are given for information rather than as the basis for regulation.

Response 22-7a-2: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule. EPA is responding to this comment because it raises issues which may be relevant to other constituents of concern in DuPont's wastes. EPA considers any exposure that lies at the 90th percentile of the distribution and greater to be at the high end of the distribution for the purpose of characterizing high end exposures. This encompasses both the 90th and 95th percentiles of the distribution. In addition, the use of a range of percentiles is made in recognition of the fact that the frequency associated with any particular level of exposure (e.g., the RfD) is uncertain and that an exposure level may occur more or less frequently than indicated by its percentile on the distribution. The point being made here is that risk management decisions are generally made based on "high end" individual risk. As a matter of ***science policy*** at EPA, the high end is defined as any exposure that lies at the 90th percentile and above (but not exceeding the highest exposure in the population). Therefore, the 95th percentile clearly qualifies as a high end estimate. For the inorganics listing, a risk management decision was made not to consider any risks above the 95th percentile, even though as a risk characterization matter, risks above the 95th percentile are just as valid an estimate of high end risks as risks at the 95th or 90th percentile.

Comment 22-7b: DuPont supports EPA's conclusions in the proposal that constituents other than manganese and thallium do not form a risk basis for the proposed listing.

Table 111-54 (65 FR 55761) and Table 111-56 (65 FR 55762) indicate that "non-wastewaters from chloride-ilmenite process" screen out or pass the risk assessment for all constituents of concern except for manganese and thallium from Iron Rich^{TM}.

DuPont supports the screening approach used by EPA as a screen and notes that comparison of SPLP or TCLP value directly to a health-based level without consideration of subsurface dilution and attenuation is not form an appropriate basis for establishing regulatory requirements.

While DuPont comments elsewhere about manganese and thallium, DuPont supports EPA's conclusion in the proposal that constituents other than manganese and thallium do not provide a risk basis for the proposed listing.

Response 22-7b: EPA acknowledges the commenter's support for its approach to screening out chemical contaminants and identifying constituents of concern. EPA notes that, although manganese was a constituent of concern in the proposal, it is deferring action on manganese-related issues in the final rule. See Section IV.B. of the preamble.

Comment 22-7c: With the appropriate manganese drinking water health-based level and reference dose, EPA's groundwater modeling would have demonstrated no manganese basis for the proposed listing.

EPA proposed to use a health-based level (HBL) for ingestion of manganese in water of 0.73 mg/L in Table 111-40 (65 FR 55752) and elsewhere in the proposal. As shown in Table 111-56 (65 FR 55762), EPA computed an HQ for manganese of 1.6 for the 90th percentile child case based on this HBL and a similarly "modified" reference dose.

Response 22-7c: EPA's responses are given below.

Comment 22-7c-1: The health-based level (HBL) for manganese in drinking water of 0.73 mg/L used in the proposal is unduly conservative and should be replaced with an HBL of 2 to 3 mg/L.

There are no promulgated primary drinking water standards for manganese. There is, however, an oral reference dose (RfD) of 0.14 mg/kg-day listed in EPA's IRIS database (EPA, 2000). As noted in IRIS, this RfD is an estimate (with uncertainty spanning perhaps an order of magnitude) considered to be safe for daily exposure to the general population (including sensitive subpopulations) over a lifetime.

For the K178 proposal, EPA used a "modifying factor" of 3 to develop a more conservative RfD for water consumption because of a number of concerns, particularly for children, expressed in the IRIS database. Further, the end notes to Table 3-3 of the Risk Assessment Background document indicate that EPA assumed a 1- to 10-year old child with a drinking water intake of 64 mL/kg/day or ~1.3 L/day to calculated the revised Rfd based on an exposure scenario that used children as opposed to adults.

As discussed below, DuPont believes that the assumptions used in calculating the HBL are inappropriate because:

- The use of the additional factor of 3 is unwarranted to protect the general population.

- Use of a child exposure is counter to the method used for calculating the safe drinking water equivalent level (DWEL) and for developing safe maximum contaminant level goals (MCLGs) under the Safe Drinking Water Act. These calculations are based on adult exposures because the levels are meant to be protective over a lifetime.

- Use of both a "modifying factor" and a child exposure is unduly conservative. The RfD is meant to be protective for the general population, including sensitive sub-populations. Using a factor to account for concerns for children and also calculating for children exposures amounts to "double counting."

Response 22-7c-1: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-7c-1a: Use of the proposed HBL is inconsistent with EPA's approach to calculated DWEL.

The IRIS database lists an oral reference dose (RfD) for Manganese as 0.14 mg/kg-day (EPA, 2000). The overall confidence in the oral RfD is medium. This value is based on EPA's review of the literature on both the toxicity of Manganese as well as its essentiality and a determination that 10 mg/day was safe for the general population. This safe level or no observed adverse effect level (NOAEL) was determined based on the following results:

- The National Research Council (1989) determined an "estimated safe and adequate daily dietary intake" of 2 - 5 mg/day.

- Others (Freeland-Graves, 1987) considered these as too low and recommended 3.5 - 7 mg/day.

- A survey of diets by WHO (1973) concludes that 8 - 9 mg/day for adults is "perfectly safe."

Assuming that the population is represented by an adult, the resulting RfD is 0.14 mg-Mn/kg body weight-day. Note that the Agency's original RfD calculations for the IRIS database utilize the traditional exposure assumptions for an adult (i.e., mean body weight of 70 kg). Then the DWEL (defined as the acceptable dose if all the exposure was from water) is 5 mg/L. As explained by Velasquez et al (1989), the DWEL is calculated based on the same adult exposure assumptions of mean body weight of 70 kg and "high-end" drinking water consumption of 2 L of water.

Use of a child scenario to calculate the HBL is inconsistent with the exposure assumptions used to calculate the DWEL on which the HBL is based.

Response 22-7c-1a: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-7c-1b: Use of a modifying factor of 3 instead of 1 is not justified.

EPA's recommendation for reducing the RfD by a "modifying factor" of 3 in the IRIS substance file for manganese (EPA, 2000) for water or soil ingestion is based on four concerns. Applying this "modifying factor" results in an RfD of 0.05 mg/kg-day which would equate to DWEL of 1.75 mg/L.

However, DuPont believes that these concerns do not merit a reduction in the RfD.

Re: Fasting individuals absorb 50% more manganese than the general population.

The results of a literature survey commissioned by EPA (Ruoff, 1995) concluded that under a wide variety of exposure scenarios in humans, the bioavailability of manganese ingested in water was essentially the same as ingested in food. The report further concluded that total diet, rather than actual medium of exposure, appeared to be the deciding factor for uptake from the gastrointestinal tract. Despite these generally accepted findings, EPA lists this as a concern for fasted individuals who appeared to exhibit a 2-fold greater uptake from water versus food.

DuPont contends that the RfD takes into account the variability in the general population and therefore, inclusion of an additional safety factor for differences of uptake between food and water is not warranted.

Re: Levels of manganese in infant formula when combined with water may pose a concern.

Although EPA concedes that no toxicity has been demonstrated, EPA's major concern appears to be for infants and the effect that manganese in infant formula and water would have on their development. If the safe level is assumed to be 0.14 mg/kg-day, then a child-specific DWEL would be calculated as 1.37 mg/L. This assumes a child less than 1-year is protective of infants and 90th % of this population consume 0.65 L/day or 102 mL/kg-day (EPA, 1997).

Infant formula does contain manganese at levels higher than in breast milk, largely to make up for the fact that manganese in soy formula is less bioavailable than manganese in breast milk (Davidsson, 1995). A review of infant formula currently marketed in the U.S. shows the quantity of manganese in infant formula ranges between 5 and 30 ug per 5 oz serving. This amounts to about 200 ug/day and turns out to be a minor contribution to an acceptable intake from water. Since infants consuming formula made from powdered formula get most, if not all, of their diet from infant formula made with water, the Relative Source Contribution (RSC) for water could be 100% and still be safe. That is, with a safe level of 1.37 mg/L, and assuming the infant drinks as much as 0.65 L/day (90%), the contribution from infant formula is about 4 times less than the total safe level from water, especially in light of the lower bioavailability of manganese in formula.

Thus, total diet consisting of water and infant formula does not warrant a reduction in the acceptable level of manganese water concentration.

Re: Neonates absorb more manganese from the GI tract and are less able to excrete manganese.
EPA suggests that there is some evidence that neonates absorb more from the gastrointestinal tract and are less able to excrete manganese and in neonates the absorbed manganese more easily passes the blood-brain barrier. However, even at a 100% retention (as calculated above), the contribution to the total diet of infants from infant formula is about 4 times less than that considered safe from drinking water.

Actual studies with human neonates have demonstrated that high plasma levels of manganese in neonates are maintained regardless of the level of manganese intake (Wilson et al, 1992). This indicates that the body's mechanisms are calibrated to retain manganese at a time when its essentiality for proper development could be critical, affirming its importance to infants and children. Thus, contrary to the situation with many chemicals, sufficient knowledge of manganese exists to conclude that no adjustment of oral exposure limits need be made to protect infants and children.

Further, two minerals common in infant formula work against the complete absorption of Mn. Calcium has been found to limit the uptake of Mn (as demonstrated in pre-term infants given Ca supplements) and excess manganese can be a problem for low iron status infants. Both of these nutrients are present in infant formula to counterbalance absorption of Mn.

It should also be noted that in the study by Kuwamura, et al (1941), although frank effects were observed in older individuals at levels of about 28 mg/L, children appeared to be unaffected.

Thus, consideration of manganese retention in infants does not warrant a reduction in the water concentration.

Re: Possible adverse effects at 2.3 mg/L in drinking water based on a single study of older individuals. Finally, EPA cites the study of Kondakis et al (1989) that raised concern for possible adverse effects associated with a lifetime consumption of drinking water containing about 2 mg/L manganese. This study was performed with only older adults (50+ years) who also had manganese in the diet of at least 5-6 mg/day (and possibly more) based on their high consumption of vegetables. Manganese in drinking water ranged from 0.36 to 2.3 mg/L in three separate areas. A battery of neurological symptoms were assessed and correlated with blood Mn and Mn in hair.

The limitations of the study precluded its use to determine a quantitative dose-response relationship. DuPont contends that taking into account that the Rouff (1995) evaluation showed no significant bioavailability difference between water and food ingestion, the combined intake of 7 - 8 mg/day is within the norm of expected intake. The neurological symptoms observed are not specific to manganese and, indeed, are typical of other symptoms of aging. The differences noted with neurological symptoms in an older population are likely to have other root causes.

A study by Vieregge et al (1995) directly contradicts the findings by Kondakis. This study found no differences on neurologic impacts of chronic oral intake of manganese in well water between subjects exposed to maximum manganese concentrations in well water ranging from 0.3 - 2.16 mg/L and those in the control group, exposed to manganese concentrations less than 0.05 mg/L.

The results of the Kondakis study do not warrant a reduction in the RfD.

On the basis of all the available information, a modifying factor of 3 is not warranted. The human system is clearly capable of easily controlling absorption and retention from daily intake amounts that range from 2.5 to more than 9 mg per day, the generally accepted range of normal intake for adults. There are no differences between absorption from food versus water (Ruoff, 1995).

Response 22-7c-1b: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-7c-1c: An HBL of 2 to 3 mg/L for manganese in drinking water is protective.

Based on the preceding discussion, the appropriate RfD for both diet and water should be the same, 0.14 mg/kg-day. Similarly, this RfD is protective of all population groups including children.

Based on this RfD, a DWEL of 4 (based on 90% adult exposure assumptions: 34 ml/kg-day, EPA, 1997) to 5 (exposure assumption consistent with the RfD development: 70 kg adult drinking 2 L/day) mg/L is appropriate. Depending on the situation-specific conditions (e.g., relative contribution from other sources), a safe, health-based limit (HBL) may be determined from this DWEL.

An HBL value of 2 to 3 mg/L in water (assuming typical intakes in food of 2-8 mg/day) appears to be appropriate.

[See original comment for references]

Response 22-7c-1c: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-7c-2: An RfD of 0.14 mg/kg-day for manganese should be used in the exposure assessment calculation of hazard quotient.

EPA presents results of hazard quotient (HQ) calculations in Table 111-56 (65 FR 55762). Section 5 of the Risk Assessment Background Document review how the HQs were calculated.

For the same reasons cited in section 7.2.1 of these comments, the modifying factor of 3 should not be used in the exposure assessment calculations and, instead, the ADD should be based on the RfD of 0.14 mg/kg-day.

Response 22-7c-2: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-7d: DuPont's planned beneficial uses of Iron Rich™ are restricted to landfill cover, berm construction, and fill applications in an industrial corridor adjacent to the Edge Moor site substantially smaller than the area modeled by EPA.

In the proposal (65 FR 55761), EPA states the following:

> The Delaware facility asserts that there are a variety of end uses for the Iron Rich™. The predominant recent use has been for the construction of dikes to contain dredged river sediments at the U.S. Army Corp of Engineer [sic] disposal sites in the vicinity of

> the titanium dioxide plant.

On the same page of the proposal, EPA goes on to say, "The Iron Rich™ has also been used as daily cover at a municipal landfill (demonstration project) and as final cover for a closed on-site landfill." EPA continues, "Other proposed uses include uses 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 the closed on-site industrial landfill."

Furthermore, the proposal (65 FR 55762) notes and the Risk Assessment Background Document (p. 2-6) confirms that EPA assumed that Iron Rich™ would be used across a 100 mile radius around the Edge Moor plant in setting up their groundwater modeling.

DuPont believes that EPA overstates the range of planned uses for Iron Rich™ in the proposal and that the Agency has erred in modeling such a large geographic area.

Response 22-7d: At the time of the proposed rule, we had concerns regarding DuPont's claims of potentially using its Iron Rich™ as a soil amendment either on-site at the Edge Moor, DE facility or within close proximity of the facility (*i.e.*, in the industrial corridor adjacent to the facility). These concerns were based upon sampling and analytical data collected by EPA and documenting the presence of manganese, thallium, and dioxins and furans in the waste at concentrations of concern. We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. It is not a constituent of concern for purposes of the final rule. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

In addition, information obtained from Delaware State (DNRCC) officials prior to proposal indicated that a previous demonstration project designed to test the adequacy of the Iron Rich™ material for use as landfill cover material resulted in a conclusion that the Iron Rich™ is too light for use as a landfill cover material. Note, however, that DuPont submitted documentation from the Delaware Solid Waste Authority (DSWA), operator of the landfill, as well as from the DSWA contractor that provides the DSWA landfill with intermediate and daily cover material, that there were in fact no issues with the successfulness of the trial demonstration project for *stabilized* Iron Rich™.[14] It is unclear whether all Iron Rich™ potentially targeted for use in this manner would be stabilized prior to placement on the land. Our data represented samples of un-stabilized Iron Rich™.

DuPont also indicated in its comments that Iron Rich™ would be used by the Army Corp of Engineers (COE) to build berms around the perimeter of a dredge disposal area at the DSWA Cherry Island Landfill. DuPont claims in its comments that "use of an Iron Rich and power plant

---

[14] Letter from Gregg W. Martin, DuPont, to Lillian Bagus, EPA, dated March 26, 2001, regarding "Inorganic Chemical Manufacturing Waste Proposal Iron-Rich™ Use and Release of VFL Documentation from CBI.

coal ash mixture has been demonstrated in the initial stages of construction for this berm." However, in conversations with EPA staff, an official from the Army Corp of Engineers indicated that the COE has no plans to use Iron Rich™ in the construction of berms or dikes. The COE official told EPA that in a previous demonstration project undertaken by the COE, the COE found that the Iron Rich™ material did not promote the growth of vegetation. The COE's specifications for berm construction materials include a requirement that the material promote the growth of vegetation. In addition, requests by the COE for additional information on the concentrations of toxic metals in the Iron Rich™ went unanswered by DuPont. Therefore, the COE has no plans at this time to use Iron Rich™ for berm construction.

Most importantly, we note that after the close of the comment period, DuPont officials indicated in an April 3, 2001 meeting with EPA staff[15] and subsequently described in an April 16, 2001 letter[16] that the company has decided not to market the Iron Rich™ material in its current composition as a soil substitute or soil amendment. DuPont officials indicated that data collected by DuPont and subsequent risk screening analyses show that the Iron Rich™ contains dioxin and furans at concentrations of concern. DuPont indicated that the company is taking steps to mitigate off-site releases of the Iron Rich™ stored at its Edge Moor facility and is investigating available off-site treatment and disposal alternatives for its newly generated Iron Rich™.

Based on this information, we believe that our off-site disposal scenario continues to be relevant, and in fact, much more likely than the scenario suggested by DuPont in their comments. DuPont has indicated that it is considering off-site landfills located as far away as South Carolina.

Comment 22-7d-1: Use of Iron Rich™ as a substitute for soil fill conserves Delaware's mineral resources.

Hydrology, Geology, and Mineral Resources of the Coastal Zone of Delaware (University of Delaware Water Resource Center, 1976) states, "Sand and gravel are the most important mineral resources in Delaware." (p. 24)

Iron Rich™ or a mixture of Iron Rich™ and power plant coal ash can be used in place of soil used for the applications described in section 5.5.5. These uses of Iron Rich™ conserve mineral resources, enabling their use in other applications.

Additionally, the manganese content of Iron Rich™ (approximately 1%) is in the range of the high end of manganese levels in uncontaminated soils in the eastern United States (0.7%).

Response 22-7d-1: As indicated in the response directly above, the use of Iron Rich™ as a soil

---

[15] "Summary of Meeting Between EPA's Office of Solid Waste and Representatives from DuPont, April 3, 2001."

[16] Letter to Ms. Lillian Bagus, U.S. EPA Office of Solid Waste from Gregg W. Martin, DuPont, April 16, 2001.

substitute is not currently viable. Due to the levels of dioxin and furans in the waste, DuPont officials have indicated (subsequent to submittal of these comments) that DuPont will not market the waste as a soil substitute in its current composition.

In addition to the dioxin risks identified by DuPont, risk assessment analyses conducted by EPA indicate that the combined Iron Rich$^{\text{TM}}$ mixture may pose significant risks to human health and the environment based upon the concentration of thallium in the combined wastestream. In today's final rule, we are listing as hazardous one component of the combined waste, the ferric chloride filter solids, based upon our assessment of the risk associated with the leachable levels of thallium in the waste. Given this, we disagree with DuPont's conclusion (as stated in its comments above) that Iron Rich$^{\text{TM}}$ is an adequate or viable substitute for soil or mineral resources.

<u>Comment 22-7d-2:</u> DNREC use and marketing regulatory program limits where Iron Rich$^{\text{TM}}$ can be used. Section 2C(6) of the Delaware Regulations Governing Solid Waste state

> For recycling solid waste into specific market applications, written approval must be obtained from the Department prior to commencing this activity.

The Department of Natural Resources and Environmental Control (DNREC) vigilantly reviews, limits, and monitors uses of Iron Rich$^{\text{TM}}$ under this regulation.

<u>Response 22-7d-2:</u> We agree with the commenter. Section 2.C.6 of the Delaware state regulations governing solid waste provides an exemption from the solid waste regulations for the "recycling of solid wastes into specific market applications." Written approval from DNREC must be obtained prior to commencing the recycling of solid wastes. To obtain approval for a recycling activity, a person must submit a written plan of operation to DNREC describing the types and quantities of materials that will recycled, the processing methods and equipment that will be used and the products that will be produced. The party proposing the recycling activity also must document the existence of markets for the product made from the recycled material.

However, we note that, as stated above, subsequent to submission of these comments, DuPont officials indicated in an April 3, 2001 meeting with EPA staff and subsequently described in an April 16, 2001 letter that due to company concerns regarding the potentially high levels of dioxins and furans in the Iron Rich$^{\text{TM}}$, DuPont does not plan to market the Iron Rich$^{\text{TM}}$, as it is currently formulated, for use as a soil substitute. In addition, the company is taking necessary precautions to mitigate potential off-site releases of the material.

<u>Comment 22-7d-3:</u> Market forces limit how far Iron Rich$^{\text{TM}}$ is likely to be transported for use.

Iron Rich$^{\text{TM}}$ competes with fill for its planned uses. The current price of fill in northern Delaware is $8/ton. The projected market price of Iron Rich$^{\text{TM}}$ is $5/ton. Local bulk truck transportation for Iron Rich$^{\text{TM}}$ has been quoted as follows:

- $2.50/ton for the first loaded mile,

- $0.10/ton from 1 to 8 loaded miles,
- $0.09/ton from 8 to 16 loaded miles, and
- $0.08/ton from 9 to 24 loaded miles.

The price differential between Iron Rich^{TM} and the competitive material dictates the break even point that the market will bear for shipping Iron Rich^{TM} to a site rather than using fill likely to be available nearby. This differential is $3/ton.

Therefore, this freight-logical evaluation indicates that Iron Rich^{TM} is likely to be shipped no more than 5 miles beyond the first loaded mile (= 5*$0.10 + $2.50) for a total of 6 miles from the Edge Moor plant where Iron Rich^{TM} is generated.

<u>Response 22-7d-3:</u> This issue appears to be moot because DuPont has abandoned plans to market Iron Rich^{TM} as described above. To the extent that it is still relevant, we disagree. In its limited cost analysis, DuPont, in calculating its "break even" price for Iron Rich^{TM} neglects to account for the savings that the company will incur by avoiding disposal costs for the Iron Rich^{TM}. If DuPont can sell the Iron Rich^{TM} it not only will gain revenue from the sale of the Iron Rich^{TM}, the company will incur a savings equal to the cost it would have incurred, had the company had to dispose of the material. Therefore, DuPont's "break even" price (or alternatively, its break even alternative waste management cost) actually is much higher than the cost/price shown by DuPont. In fact, DuPont should be willing to "sell" its Iron Rich^{TM} at a loss (or actually pay customers to take the Iron Rich^{TM}), as long as the loss (or price) to DuPont is less than the price that DuPont would incur to dispose of the material (or the lowest cost alternative management).

In conversations with officials at DNREC, DNREC staff indicated that the current tipping fee for a ton of solid waste at a DSWA landfill is $58.50.[17] (Tipping fee provided is for a municipal solid waste landfill.) Therefore, DuPont's economic "break even" point (i.e., the price the company is willing to pay to discard the material or loss at which the company is willing to sell the Iron Rich^{TM}) is much higher than the price presented by the company. DuPont could economically ship the material a much further distance and still incur a substantial savings over the waste management costs the company may incur to dispose of the material.

In fact, DuPont should be willing to incur costs of close to $58.50/ton to ship the Iron Rich^{TM} material to customer locations for use as a soil (or fill) substitute. Using DuPont's cost figures, DuPont should be willing to incur transportation costs of close to $51.00/ton ($58.50 - $5.00/ton "market price" - $2.50/ton fixed price for first mile of transport). At 8 cents per "loaded mile" that would allow DuPont to transport the Iron Rich^{TM} over 600 miles before incurring costs greater than the cost associated with the alternative management option (landfilling). Actually, in the case of customers located closer than 600 miles, DuPont may be willing to both incur the transportation

---

[17] Phone conversation between Matthew Higgins, DNREC and Patricia Overmeyer, US EPA, January 4, 2001.

cost and pay the customer a "tipping fee" to take the Iron Rich^{TM}, as long as the total cost to DuPont, per ton of Iron Rich^{TM}, is less than $58.50/ton (or the associated cost for another alternative management option).

Comment 22-7d-4: Mixing with power plant coal ash prior to use is expected to reduce metals leachability.

For most uses, Iron Rich^{TM} is marketed as a blend with coal fly ash. The blend is nominally 70% Iron Rich^{TM} and 30% coal fly ash.

In addition to the structural benefits of adding coal fly ash to Iron Rich^{TM}, the alkaline nature of coal ash tends to reduce metal mobility. Many studies have shown that metals are less likely to migrate at higher pH. For example, manganese sorption on hydrous ferric oxide is predicted via Dzombak and Morel's Generalized Two-Layer Model to increase by 3 orders of magnitude from pH 7 to 9. Therefore, mixing with coal fly ash prior to use is expected to significantly reduce metals leachability.

Response 22-7d-4: As stated above, DuPont officials indicated in a meeting with EPA staff that due to company concerns regarding the potentially high levels of dioxins and furans in the Iron Rich^{TM}, DuPont does not plan to market the Iron Rich^{TM}, as it is currently formulated, for use as a soil substitute. In addition, the company is taking necessary precautions to mitigate potential off-site releases of the material. Therefore, the possible reduction in metal leachability when mixed with fly ash is not relevant to our final listing decision. We note that we are deferring final action on all elements of our proposal that are specifically related to the waste constituent. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule. We are responding to this comment because it may be relevant to other metal constituents of concern in the ferric chloride solids.

Comment 22-7d-5: Planned Iron Rich^{TM} uses are limited to an industrial corridor approximately 1 mile wide along Delaware River and 5 miles south of the Edge Moor plant.

The locations for planned use of Iron Rich^{TM} are limited to a very specific industrial corridor shown in Attachment 7.4.5-A. These four locations, from north to south, are labeled:

- Current Iron Rich^{TM} Staging Area,
- DSWA Cherry Island Daily Cover,
- COE Cherry Island Berm, and
- COE Harbor South.

This industrial corridor is approximately 1 mile wide and 4 miles long. It is bounded by Route 495 to the west and the Delaware River to the east. The northern end of this corridor is the DuPont Edge Moor Plant. The southern end of the corridor is approximately four miles south of the DuPont Edge Moor Plant and is bounded by the closed DSWA Pigeon Point

Landfill (which, in turn, is north of the Delaware Memorial Bridge).

New Castle County Zoning regulations classify this entire corridor as HI (heavy industrial) and thereby limit this corridor to industrial uses.

Response 22-7d-5: As stated in our responses to comments above, DuPont has not demonstrated that the use of Iron Rich^{TM} as a berm construction material is viable. In addition, DuPont officials have indicated that the company is concerned about the levels of dioxins and furans in the waste and therefore no longer plans to market the material as a soil substitute or soil amendment material, as currently formulated. Therefore, the commenter's arguments regarding the location and size of the industrial corridor and the zoning classification of the area are moot.

Comment 22-7d-5-1: Current Iron Rich^{TM} Staging Area

DuPont currently stages Iron Rich^{TM} on DuPont property next to its closed Cherry Island landfill prior to shipment to off-site use locations. This area, like the rest of Cherry Island, is underlain by approximately 30 feet of predominately silt and clay dredge spoil material with a permeability of 10-5 to 10-7 cm/sec. This location is bound by Shell Pot Creek to the north and the Delaware River to the east.

This staging area is approximately 25 acres and contains approximately 500,000 tons of Iron Rich. The area is access-controlled with fencing.

Following resumption of other uses for Iron Rich^{TM} (described elsewhere in section 7.4.5), DuPont plans to use to discontinue use of this staging area. One of the options being evaluated for this area is to use the Iron Rich^{TM} in this area as fill material for contour management as part of developing this area into a natural vegetated area.

Response 22-7d-5-1: EPA is listing as hazardous one component (i.e., the ferric chloride filter solids) of the combined Iron Rich^{TM} currently stored in the on-site staging pile. Given that the Iron Rich^{TM} contained in the staging area contains the ferric chloride filter solids, use of the material as fill material for contour management and as part of a natural vegetated area after the effective date of the final rule, if carried out after the effective date of the listing rule, will have to comply with applicable RCRA hazardous waste regulations. The staging area will ultimately be closed pursuant to the applicable waste regulations in the State of Delaware.

Comment 22-7d-5-2: DSWA Cherry Island Landfill

The Delaware Solid Waste Authority (DSWA) is an agency which is empowered by State law to manage all the solid waste generated in Delaware's three counties.

In New Castle County, the DSWA operates the Northern Solid Waste Facility II, commonly referred to as DSWA Cherry Island. (This landfill is independent of and is not affiliated with the closed DuPont Cherry Island Landfill.) The DSWA Cherry Island Landfill has restricted access with fencing and entry monitoring.

This landfill is further described in a DSWA pamphlet included as Attachment 7.4.5-B.

Daily Cover

This operating municipal solid waste (MSW) landfill requires 6 inches of daily cover over the working face of the landfill. A mixture of 70% Iron Rich™ and 30% power plant coal ash is planned for use for this daily cover. This use is expected to utilize approximately 20,000 tons of Iron Rich™ each year. Use of an Iron Rich™ and power plant coal ash mixture for this purpose has already been demonstrated.

This DSWA landfill has an engineered liner with a permeability of 10-7 cm/sec and a leachate collection. Leachate collected from this landfill is treated in the City of Wilmington wastewater treatment plant. Closure of the existing cell(s) is expected to occur in 2010. Upon closure, a capping system will be installed on this landfill in accordance with Section 5.H of Delaware Regulations Governing Solid Waste, which are consistent with the federal criteria in 40 CFR 258.

Berm Construction

A berm/dike is being built around the perimeter of the current Corps of Engineers (COE) dredge disposal area. This dredge disposal area is in an area southwest of, and contiguous to, the DSWA Cherry Island Landfill. Use of an Iron Rich™ and power plant coal ash mixture has been demonstrated in the initial stages of construction for this berm.

Iron Rich™ is planned for use with natural soils to construct the perimeter berm walls around an area bounded by Route 495 to the west and the Christina River to the South.

The area inside the berm will periodically be filled with dredge spoils from the Delaware River and other local waterways that the COE is responsible to maintain. The berm will be raised every few years as the silt and clay spoil material increases in depth inside.

This use of Iron Rich™ is expected to utilize approximately 60,000 tons per year.

When the current DSWA Cherry Island Landfill is closed, this berm/dredge spoil area is expected to become the expansion area for the DSWA Cherry Island Landfill, which will have an engineered liner with a permeability of 10-7 cm/sec, a leachate collection and treatment system. This area, like the rest of Cherry Island, is underlain by approximately 30 feet of predominately silt and clay dredge spoil material with a permeability of 10-5 to 10-7 cm/sec. Upon closure, a capping system will be installed on this landfill in accordance with Section 5.H of Delaware Regulations Governing Solid Waste, which are consistent with the federal criteria in 40 CFR 258.

Presuming that Iron Rich™ would also be used as daily cover for this future expanded DSWA Cherry Island Landfill, the overall area of this use (including the berms) would be approximately 300 acres.

Response 22-7d-5-2: We thank the commenter for the additional information and explanation of its planned uses for the Iron Rich™ material. We note that DuPont officials indicated in an April 3, 2001 meeting with EPA staff that the company no longer plans to market the Iron Rich™ material, as it currently is formulated, for these uses.

Comment 22-7d-5-3: COE Harbor South Berm Construction

The Corps of Engineers is constructing berms in an area next to the Port of Wilmington to build up a sloped area that will be paved over and used for Port operations (e.g., warehousing or parking). This area is underlain by silty clay dredge spoil material. The Port of Wilmington is located at the conflux of the Christina and Delaware Rivers, with the Port docks being located on the Christina River. As part of the Port of Wilmington site, Harbor South has access controls including fencing and manned gates.

Iron Rich™ is planned for use in constructing these berms. Use of an Iron Rich™ and power plant coal ash mixture has been demonstrated in the initial stages of construction for this berm.

Berm construction would utilize approximately 20,000 tons of Iron Rich™ per year. Once complete, the berms are expected to cover a total area of approximately 30 acres.

Response 22-7d-5-3: See response to comment 22-7d-5-2.

Comment 22-7d-6: The geologic setting of the Iron Rich reuse industrial corridor provides confinement.

Comment 22-7d-6-1: The area is underlain by coastal plain sediments and bedrock.

The Iron Rich reuse industrial corridor is located in northern New Castle County, Delaware. The geological province for this area is the Atlantic Coastal Plan (Sundstrom and Picket, 1971). The sedimentary formation of the Coastal Plain is underlain by the bedrock of the Appalachian Piedmont that outcrops to the west.

In this area, the thickness of the Coastal Plain sediments is typically less than 400 feet around the Delaware Memorial Bridges and less than 200 feet in the area of Cherry Island. Just north of the DuPont Edge Moor Plant, DGS log Bd55-07 shows bedrock at a depth of 84 feet below ground surface. See Attachment 7.4.6-A for and other DGS (Delaware Geological Survey) logs.

The Coastal Plain sediments consist of two geological formations. On top of the bedrock lies the Cretaceous age (75 million year old) Potomac Formation. On top of the Potomac Formation lies the Pleistocene age (1 million year old) Columbia Formation. On top of the Columbia Formation, close to the banks of the Delaware River, is a recent age (i.e. last 10,000 years) deposit of peat material with some silt and clay.

The following maps and reports support the above interpretation:

C *The Availability of Ground Water in New Castle County Delaware,* R. W. Sundstrom and T.E. Pickett, 1971, University of Delaware Water Resources Center, Newark, Delaware, p. 156.

C *Geology of the Wilmington Area, Delaware,* Kenneth D. Woodruff and Allan M. Thompson, 1975, Delaware Geological Survey, Geologic Map Series, No. 4., p. 1

C *Columbia (Pleistocene) Sediments of Delaware,* R.R. Jordan, 1963, Delaware Geological survey Bulletin 12, Newark, Delaware.

Response 22-7d-6-1: EPA thanks the commenter for directing its attention to this information on the geology of the Cherry Island area. We note that DuPont officials indicated in an April 3, 2001 meeting with EPA staff that the company no longer plans to market the Iron Rich™ material, as it currently is formulated, for these uses. To the extent that the information is still relevant, we do not completely agree with DuPont's position. In agreement with the DGS boring log cited by the commenter, the Columbia (Pleistocene) sediments are shown on the Woodruff and Thompson map (1975) as being around 100 feet thick at Cherry Island. The reports referenced also indicate that in general the Columbia aquifer is regionally considered unconfined in New Castle county. Therefore the only significant hydraulic confinement of the Columbia aquifer in the Cherry Island area would be the Holocene deposits (including the dredge spoil) near the river. These natural sandy silts/clays above the Columbia may also exhibit semi-confining properties in conjunction with the peat layer where present.

However, it is not clear that a semi-confined condition is laterally continuous. Since the Holocene/spoil sediments are fully saturated beginning at a shallow depth below ground, the Columbia aquifer may be more unconfined where the peat is absent. Further, there exists a shallow sand aquifer generally in a zone from approximately 13 feet below ground surface to 23 feet below ground surface. Though probably not extensive, this sandy layer appears to be laterally continuous in the corridor. It is conceivable that a shallow well might withdraw water from this layer. There are many wells in the well information supplied by the commenter that are less than 30 feet deep. For this reason, EPA considers the 20-foot sand to be a candidate for possible ground water modeling.

Conceptually, the hydrogeology of the area as suggested by the data is one in which the opportunity exists for contamination to infiltrate about 5 feet of unsaturated sand, silt, and clay layers of varying textures. The upper saturated zone (to ~13 feet below ground surface) consists of a sandy silt/clay that grades into a more sandy zone (20-foot aquifer) with interbedded lenses and fine layers of fine to medium sand (to ~23 feet below ground surface). It is likely that the 20-foot aquifer is locally semi-confined. Available elevation data suggests that ground water flow in this unit would most likely discharge to the Delaware River. A significant peat and clay layer (approximately 5 plus feet thick) rests between the 20-foot aquifer and the Columbia Aquifer. Although the Columbia Aquifer is generally considered to be unconfined in most areas of northern Delaware, the water-level data suggests that it may be hydraulically semi-confined in the area of the corridor. In particular, there appears to be a significant hydraulic separation (23 feet) between the water level in the shallow aquifer and the piezometric levels in the Columbia Aquifer.

<u>Comment 22-7d-6-2:</u> Low permeability dredge spoils provide a low permeability layer between Iron Rich uses and the Coastal Plain sediments.

The sediments of the Coastal Plain and the peat unit are overlain by a thick (30 feet or more) blanket of hydraulically in-placed dredge spoils in all the areas proposed for Iron Rich reuse projects. This has been officially mapped by the DGS in a 1984 map by Kenneth D. Woodruff of the DGS, entitled *Geohydrology of the Wilmington Area, Delaware.* The thickness of this surficial dredge spoil unit has been documented with hundreds of soil borings in the area. DuPont alone has drilled over 60 soil borings on their Cherry Island property. A sample of these logs are included in Attachment 7.4.6-B.

A summary of the ASTM testing for hydraulic conductivity and particle size distribution for the dredge spoil material found on the DuPont property is found in Attachment 7.4.6-C. As can be seen from the attachment, the hydraulic conductivity, K, is typically 10-5 to 10-7 cm/sec and the particle size is typical of clay and silt. Data is also provided for the Peat unit, which also has a K of 10-' cm/sec and is typically 10 feet thick. The Delaware Solid Waste Authority intentionally located their MSW landfills (Pigeon Point and DSWA Cherry Island) over these spoils and the peat unit because of their excellent confining properties.

<u>Response 22-7d-6-2:</u> We note that DuPont officials indicated in an April 3, 2001 meeting with EPA staff that the company no longer plans to market the Iron Rich^{TM} material, as it currently is formulated, for these uses. To the extent that the comment is still relevant, we disagree. The boring logs provided by the commenter show materials within the upper 30 feet that are similar to materials characteristic of dredge spoils. However, it is unclear if these materials consistently represent dredge spoils or they might also represent naturally deposited Holocene sandy silt/clay layers. (The map entitled *Geohydrology of the Wilmington Area, Delaware* referred to by the commenter was not available to EPA to review.) If the shallow 20-foot sand layer noted above was naturally deposited, this might imply that the areal extent of the shallow sand could be significant.

The range of hydraulic conductivities attributed to the dredge spoils suggests that at least in some areas a water-table condition may be present, as opposed to the Holocene sediments/dredge spoils acting to confine the Columbia formation. The average K value calculated from the data is 1.7E-06 cm/sec (range from 4.70E-07 cm/sec to 1.10E-05 cm/sec). This average K value is representative of a silty sand. There are sieve analysis results for samples from boring T-8 but it is unclear if these are from generally the same depth interval.

EPA notes that the boring logs call into question the representativeness of the samples used for the unsaturated zone hydraulic conductivity measurements. Careful review of the lithologic data provided by the commenter supports the view that the 30 to 40 feet of silty clay (Holocene/dredge spoil) deposits over the Columbia/Potomac aquifers may not be a continuous zone of silt and clay material in all areas of the corridor. Both minor and more significant permeable sand zones are present within these materials. For example, the sand layer noted previously within which monitoring wells have been installed are noted on DuPont's and DGS's boring records. If the shallow "20-foot sand layer" is a natural Holocene deposit, rather than a product of the dredging process, its aerial distribution and hydraulic connection becomes important as a possible conduit for contaminant transport. Even though the boring records do indicate that texturally the Holocene deposits are predominantly silt and clay, the available data and documentation suggests that the sand content within the more significant clay and silt zones, above and below the 20-foot sand layer, appear to contain varying amounts of sand. Even the characteristically less permeable peat layer situated between the Holocene/dredge spoil and the sands of the Columbia Aquifer is not always present and where it occurs is often described as sandy.

This suggests that the sediments/fill material above the Columbia aquifer may exhibit a wide range of possible hydraulic conductivities and also that the Columbia/Potomac aquifers may be better characterized as truly unconfined in areas of the corridor. This more conservative characterization for the entire area of the corridor would suggest that representing the unsaturated and/or part of the saturated zone thickness as silty clay or clayey silt in general may not be appropriate. For purposes of this modeling the 30 to 40 feet of Holocene/dredge spoils may be better characterized with a somewhat higher (averaged) permeability typified by a clayey/silty sand texture rather than silty clay texture of the samples subjected to hydraulic conductivity testing.

Comment 22-7d-7: Groundwater in this area flows to adjacent surface water.

In the Iron Rich reuse industrial corridor, the "groundwater" found in the dredge spoil material seeps at low volumes and rates of travel into the nearest surface water body. A very small amount of recharge may be provided to the underlying Coastal Plain formations. These formations in-turn discharge to the Delaware River taking a deeper and longer flow path. In the area of the DSWA Cherry Island Landfill, some water seeps to the Christina River (which in turn discharges to the Delaware River) and some directly into the Delaware River. At the DuPont Cherry Island location, some water seeps to Shell Pot Creek (which drains to the Delaware River) and some directly into the Delaware River. Flow maps for each of these areas is provided in Attachment 7.4.7. DuPont has over 15 years of groundwater flow direction maps for it's Cherry Island property.

It should be noted that the DGS hydrogeological flow maps for the Wilmington/New Castle area do not map water levels north of the Delaware Memorial Bridges because there are no wells (other than shallow environmental observation wells) to provide water level data. This indicates that the Iron Rich reuse corridor area is not over a viable potable water aquifer.

<u>Response 22-7d-7:</u> EPA thanks the commenter for providing this information on the direction of ground water flow in the Cherry Island area. We note that DuPont officials indicated in an April 3, 2001 meeting with EPA staff that the company no longer plans to market the Iron Rich^{TM} material, as it currently is formulated, for these uses. To the extent that the comment is still relevant, we disagree. Although the ground water flow maps indicate that ground water flows toward the rivers from the shallow sand layers and the deeper Columbian aquifer, the maps also indicate the existence of three distinct saturated zones, including a "20-foot aquifer zone" beneath the DuPont property staging area that correlates with a zone of alternating clay/silt with seams and apparently thin layers of fine sand. This zone is generally situated between 13-ft and 23-ft below ground surface. The 20-foot aquifer zone is generally overlain by more significant clay/silt layers. The clay layer is described as moist/sticky/plastic indicating that the 20-foot aquifer zone is probably only semi-confined but may display the characteristics of a water-table condition in some areas. Therefore, the use of shallow water-levels from the 20-foot aquifer should be considered with the assumption that the Columbia Aquifer is generally unconfined.

EPA determined the water level by comparing the boring log information for the Cherry Island property with the flow maps provided for the 20-foot aquifer in the staging area. The elevation from the flow maps indicate that the water-level elevation ranges between 10 ft to 17 ft. The reference elevation for all borings (with the exception of No. 47) is given as 14.5 ft msl. This suggests a range of depths below ground surface for the water table of between 1.5 ft. (~0.5 m) and 4.5 ft (~1.4 m).

The local hydraulic gradient as measured from the flow maps ranges between 0.004 and 0.006. The gradient of the potentiometric surface for the Columbia Aquifer generally ranges between 0.0002 and 0.0008. If the Columbia is considered an unconfined aquifer, then the range of gradient values between the shallow and deeper aquifer should be considered when evaluating the velocity of ground water flow.

EPA did not attempt to conduct a detailed review of the available well information due to the nature of the information provided by the commenter. For example, many of the column headings are missing from the tabulated data. The data purportedly include all wells for any use; however, no specific uses can be gleaned from the table. Only street or post office box information is available to discern the location of the wells. Many small-diameter shallow wells are identified, but many of these are probably monitoring wells or piezometers. The large number of 2-inch and 3-inch wells listed are also commonly used in coastal plain areas for irrigation and water supply. Several 4-inch diameter wells are listed which is also a common well diameter used for domestic and commercial supply. Only one large diameter well was listed and that well appears to be located over 5 miles away. Although the closest offsite location appears to be approximately 1 mile away (across I-495), experience has shown that ground water users can be located in the most unlikely or unexpected places.

<u>Comment 22-7d-8:</u> Use at DSWA landfills provides additional controls.

The DSWA Cherry Island Landfill cells provide an excellent environmental settings for Iron Rich reuse because of engineering controls required by DNREC. The engineered liner meets a permeability standard of 10-7 cm/sec. This liner must be a minimum of 5 feet above the water table in accordance with State solid waste regulations. The landfill cells are required to have a leachate collection system. Once the DSWA Cherry Island Landfill is closed, it will also be required by regulation to have a capping system designed to minimize or eliminate infiltration.

Response 22-7d-8: EPA thanks DuPont for providing this additional information. As stated in our responses to comments presented above, information available to EPA indicates that landfill cover is not a viable use for the Iron Rich^{TM} material. In addition, DuPont officials have told EPA staff that the company no longer plans to market the Iron Rich^{TM}, as it currently is formulated, as a landfill cover material.

Comment 22-7d-9: The nearest drinking water well to planned Iron Rich uses is over 1 mile away.

The geological boring logs at the DGS also indicate water wells if installed. In addition DuPont requested a well radius search from DNREC's Division of Water Resources, Water Supply Section. The results of this search are found in Attachment 7.4.9. There is no consumptive use of water within 1 mile of any of the proposed locations for Iron Rich reuse.

The nearest municipal water system is located in Collins Park, with wells approximately 1.2 miles up-gradient or cross-gradient of the closed Pigeon Point landfill. This well field is operated by Artesian Water Company. Mr. Dan Barbato of Artesian confirms that this is their "franchise" district and no other private residential drinking water wells would be allowed by the DNREC. The water franchises in Delaware are officially called "Certificates of Public Necessity and Convenience" and are issued by the Public Utilities Commission. The DNREC Water Supply Section will not issue a residential well permit in certificated areas.

The Atlas Point industrial site located just south of Delaware Memorial Bridge has a drinking water well for consumptive use on-site.

In the area to the west of the Iron Rich reuse industrial corridor, the City of Wilmington provides all the drinking water. There water supply is a surface water intake on the Brandywine River, located north of the City. (Further downstream, the Brandywine River discharges to the Christina River which in turn discharges to the Delaware River.) The Delaware Water Resource Agency (WRA), located at the University of Delaware, have mapped the groundwater recharge protection areas and municipal water uses for New Castle County. While the map is dated 1993, the WRA confirms that there have been no changes in well-head protection areas in the area designated as the Iron Rich reuse corridor since 1993. The WRA map confirms the information provided above.

Response 22-7d-9: EPA thanks for DuPont for providing this additional information. It no longer appears relevant, since DuPont no longer plans to confine placement of Iron Rich to the area described in this comment.

Comment 22-7e: Access controls and cover materials for all planned uses prevent soil ingestion and moot consideration of dioxin soil ingestion screening levels.

In the proposal (65 FR 55763), EPA appears to say that Iron Rich^{TM} contains dioxin in a total composition concentration in excess of a 45 ppt (WHO-TEQ soil ingestion HBL (health-based limit). On the same page in the proposal, EPA goes on to say

> While we did not conduct a risk assessment of the detected TCDD TEQ, the presence of TCDD equivalents in the wastes is an additional factor that supports a listing determination, particularly in light of fact that the management practices reported by the facility were varied and in many cases, would constitute releases to the circulating environment with a greater potential for a variety of exposure pathways than would occur from a well managed landfill.

EPA's SPLP analytical results for Iron Rich^{TM} for dioxin in Table 3-27 of the *Titanium Dioxide Listing Background for the Inorganic Chemical Listing Determination* indicate that dioxin is not expected to leach out of this material and therefore would not enter the groundwater pathway.

Each Iron Rich^{TM} use described in section 7.4.5 prevents soil ingestion via access controls (e.g., fencing), landfill capping, vegetative cover material, or paving.

The management practices (especially those involving MSW landfills) described in section 7.4.5 do not constitute releases of dioxin to the circulating environment and do not have greater potential for exposure than would occur from a well managed landfill.

Response 22-7e: This comment is no longer relevant given the changes in management practices reported by DuPont in an April 3, 2001 meeting with EPA staff and a subsequent April 16th letter. These changes are directly linked to DuPont's recent assessment of risk associated with dioxin in the scenarios described above.

Comment 22-7f: With more appropriate consideration of attenuation due to metals sorption, EPA's groundwater modeling would have demonstrated no basis for the proposed listing.

In groundwater modeling for the proposal, the Agency used empirical distributions for soil-water partitioning coefficient (K_d) for metals instead of using MINTEQA2 (65 FR 55698).

While DuPont urges the Agency to reconsider use of MINTEQA2 for this rulemaking as this has proven to be a scientifically-valid model, these comments focus on the empirical approach used by EPA for this listing proposal.

In the Risk Assessment Background Document, EPA admits that it had very limited K_d data for thallium and hence used a log uniform distribution over 0 to 1000 L/kg (p. 4-72) [The median value of such a distribution is $10^{1.5}$ or 31.6 L/kg.] For manganese, EPA used a "rank-order percentile approach" of K_d values across a wide variety of soil types, apparently assuming K_d values across all soil types to be members of the same population.

<u>Response 22-7f:</u> As stated by the commenter, EPA relied on empirical data on soil-water partitioning (i.e., K_d) for use in modeling contaminant transport of metals. Although EPA has used the MINTEQA2 equilibrium speciation model to estimate K_d's, a number of technical issues have arisen concerning the model and its application. As indicated in the preamble to the proposed rule, EPA is in the process of evaluating the model to address those issues. Once the evaluation is completed and the issues are satisfactorily resolved, EPA will make a determination on the extent to which the model may be used in the future. As explained below and in the preamble, EPA modified its approach to establishing K_ds for this listing evaluation. We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule. We are responding to this comment because it remains relevant to thallium.

The other issues identified by the commenter are addressed below.

<u>Comment 22-7f-1:</u> K_d values should be assigned according to site-specific factors (e.g., soil type) rather than selected from a statistical distribution.

However, a variety of references indicate that K_d for a metal is a function of key variables, including soil type. For example, Lee et al, (ES&T, Vol 30, No. 12, 1996) states

> The mobility and fate of metals in the soil environment are directly related to their partitioning between soil and soil solution. The presence of trace metals in the solid phase is a result of precipitation and adsorption to components of the soil, processes that are highly pH dependent. For trace metals, adsorption is normally of greater importance than is precipitation. Varying contents of organic matter and metal oxides may result in varying capacity for metal sorption onto soils.

Assuming that a variety of K_d values across a number of soil types are members of the same population runs counter to statistical theory. Using a rank-order percentile or any other approach to formulating an empirical probability distribution without the values first belonging to the same population is not appropriate.

Rather, like geological and hydrogeological parameters in the EPACMTP model, EPA should base the K_d value for a given metal on the soil type and, to the extent that is known, pH.

Response 22-7f-1: It is widely known that K_d varies greatly depending on the specific geochemical conditions in the soil and aquifer environment. EPA considered this fact in collecting and utilizing the literature derived soil-water K_d values used in the ground water modeling analysis. These data clearly demonstrate that K_d may vary over several orders of magnitude. Soil type, pH, and available sorption sites affect the value of K_d and, as a result, K_d can vary widely, both from site-to-site and locally at any given site. To address this source of uncertainty, EPA used an empirical distribution based on the literature derived K_d’s.

EPA disagrees with the commenter that soil type is a good predictor of K_d. A wide range of variables can affect the adsorption of a particular metal species, as does the form of the metal species itself and the set of experimental conditions for a particular study. For example, as pointed out by Lee et al (1996), one of the most important variables, metal oxides, can be difficult to measure and is often not measured in the available literature. A simplistic relationship, based on soil type (or pH), will not consider these or other important influences on K_d. These include the number and type of sorption sites, ligand solution chemistry, and metal concentration.

In summary, EPA believes the available literature support large variability and uncertainty in K_d and that using the full range of measured variability is the best way to accommodate both the variability and uncertainty in a site-based regional analysis. However, as explained below, EPA agrees with the commenter that EPA’s K_ds overestimated mobility at low concentrations of thallium. EPA adjusted its risk assessment and revised its results for thallium. See response 22-7f-4.

Comment 22-7f-2: EPA's data set supports a manganese K_d of 430 L/kg for the Iron Rich reuse corridor.

The primary source used by EPA for manganese K_d is a Finnish paper by Miettinen, Nikula, and Leskinen from the docket for the proposed rule.

In this paper, the authors present manganese K_d data by soil type. For silty clay (the soil type representative of the Iron Rich reuse corridor), the authors present a K_d value of 430 L/kg.

Response 22-7f-2: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today’s final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-7f-3: DuPont determined K_d values for thallium to fill EPA's data void.

Given the paucity of K_d values for thallium in the Risk Assessment Background Document, DuPont contracted Centre Labs to do an experimental study to determine K_d values.

Although the report for this study will be submitted later, the results support a K_d for thallium for a soil representative of the soil in the Iron Rich reuse corridor of 500 L/kg.

Response 22-7f-3: EPA thanks the commenter for providing the data on K_d for thallium. EPA reviewed the report submitted by the commenter and concludes that the study was well designed and implemented and was generally done in accordance with available guidance for conducting such studies. EPA notes that the Center Labs data are consistent with the range of K_d's EPA used in the risk analysis for the proposed rule, given the limited range of soils tested.

EPA does not agree with the commenter that a single value of 500 L/kg is supported by this study. The suggestion that the study supports a K_d of 500 L/kg for thallium appears to be based on the data reported for the Baptistown soil. The K_d for this soil ranges from 800 L/kg at the lowest thallium concentration tested (initial concentration 0.25 mg/L) down to 50 L/kg at the highest concentration (initial concentration 10 mg/L). This suggests that the full Freundlich isotherm should be used rather than a single value for K_d. (EPA notes that other information submitted by the commenter indicates a range of K_d's were considered, i.e., values of 100, 150, 200, 225, and 500 L/kg were used by the commenter in subsequent modeling analyses for thallium).

Comment 22-7f-4: EPACMTP modeling, conducted with the appropriate K_d values show very high dilution-attenuation factors for thallium and manganese and therefore very low HQs.

With appropriate input parameters, EPACMTP is an appropriate groundwater modeling tool for risk assessment.

For a K_d value of 500 L/kg for thallium, EPACMTP yielded a dilution-attenuation factor (DAF) on the order of 10^7 rather than the DAF of 7 obtained by EPA.

For a K_d value of 430 L/kg for manganese, EPACMTP yielded a DAF on the order of 10^7.

Therefore, with such high DAFs, the hazard quotients for thallium and manganese would be well below 1.

Response 22-7f-4: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule. We are responding to this comment because it is relevant to thallium. EPA does not agree that using a single K_d value is appropriate, for the reasons stated previously and, therefore, does not agree with the conclusion that the modeled DAF's support hazard quotients below 1. (EPA notes that other information submitted by the commenter indicates a range of K_d's were considered, i.e., values of 100, 150, 200, 225, and 500 L/kg were used subsequently by the commenter in modeling analyses for thallium. However, EPA notes further that for thallium, the Center Labs study found higher K_d values at lower concentrations. Considering the relatively low concentrations of thallium in the waste, EPA concludes that the risk analysis at proposal likely underestimated the extent of attenuation attributable to sorption and, therefore, overstated the risks from thallium. Therefore, EPA agrees with the commenter that hazard quotients for thallium would be below 1, given the Center Labs data.)

<u>Comment 22-7g:</u> With site-specific factors for actual planned, restricted uses of Iron Rich^{TM} groundwater modeling does not support hazardous waste listing.

The modeling that EPA performed was not representative of the locations in which Iron Rich will be used. Additional modeling using more representative parameters was performed using EPACMTP.

It must be noted that the DSWA landfills where the majority of Iron Rich will be used will have engineered liners and leachate collection systems as described in Section 7.4 which will prevent any migration of leachate, and, hence, there will be no exposure via groundwater. Also, groundwater in the upper most aquifer (the Columbia Formation) which may potentially receive leachate discharges to surface water bodies, and therefore a groundwater well as a receptor is extremely unlikely. Therefore, the EPACMTP modeling described below is essentially representative of worst case conditions since the above factors are not considered.

The modeling effort focused on the deterministic high-end parameter analyses for the ilmenite waste water treatment solids from the DuPont Edge Moor Plant described in USEPA's *Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes: Background Document* (EPA, 2000). The species of interest were manganese and thallium. A site-specific conceptual model was developed and a series of simulations were performed to evaluate the applicability of USEPA's results when applied to the proposed sites where Iron Rich will be used.

The EPA provided their input files to use as a starting point in this study and for comparison with DuPont results. Although the input file parameters did not always match the parameters described in the background document, the results generated did. It should also be noted that where the input files differed from the background document listing of parameters, the parameters as found in the input files were used.

Parameters of note that differed between the input files and the document included the choice of high-end parameters for thallium, the thallium central tendency K_d, and the K_d table for manganese. The thallium high-end parameters for the DuPont Edge Moor Plant as described in the background document and its appendices were the x-well distance and the intake rate. The high-end parameters used in the simulation were the landfill area and the y-coordinate of the well ($y = 0$). The thallium central tendency K_d as listed in the document is 500 cc/g as opposed to the value of 31.6 cc/g as found in the data set. Since 31.6 is the median of the log-uniform distribution described in the document this value was used. The K_d table for manganese listed in the background document has one less term than the table found in the input file. The central-tendency values also differ with the document (see Table 4-25). The document indicated 12 values and showed a value of 144 cc/g and the input file had 13 values and a value of 113 cc/g as noted in Table 4-68.

The site-specific data used to develop the conceptual model for the simulations represent the industrial corridor in which Iron Rich will be used (see Section 7.4). The dredge spoils and peat zone material is a silty clay 30 to 40 ft thick (modeled as 9.14 meters) with measured hydraulic conductivity values ranging from 10^{-5} to 10^{-7} cm/sec (see Section 7.4). This material was conservatively modeled as 10^{-5} cm/sec. These materials are underlain by a sand (Columbia formation) with a tested hydraulic conductivity value of 0.8×10^{-3} cm/sec (252 m/yr). The thickness of the sand was assumed to be 10.7 meters. The potential migration pathway is downward through the silty clay into the sand and then horizontally through the sand. EPACMTP does not consider heterogeneity in the saturated zone, so this scenario was approximated by assuming that the silty clay was the unsaturated zone. Since the minimal K_ds being considered were over 30 cc/g, the influence of saturation (and porosity) on retarded transport time will be negligible. An infiltration and recharge rate of 0.1641 m/year that was used in EPA's modeling for silty clay loam is similar to the dredge spoils and was also used.

The three separate waste units considered were

1. a combination of both the DWSA Cherry Island Daily Cover and COE Cherry Island Berm (since they are essentially the same area),
2. COE Harbor South, and
3. the DuPont Edge Moor Current Iron Rich Staging Area.

The sizes and waste quantities for each of these sites are presented in the table below. The size of the area of waste placement was scaled off of maps, and the waste quantity was based on the estimated annual usage multiplied by 30 years. The thickness of waste was calculated for input to the model based on the total mass of material after 30 years and the bulk density. A conservative well distance of 1600 m was selected based on topographical and physical constraints (e.g., rivers, highways, etc.) for each of the sites. Distances to the nearest well used for municipal or industrial drinking water are much further.

Area	Length, meters	Width, meters	US tons/yr	Number of years	Total US Tons	Density kg/1	Area, sq. meters	Depth of Waste, meters
DSWA Cherry Island berms/ cover	1,500	1,000	80,000	30	2,400,000	1.5	1,500,000	0.97
Port of Wilm. Harbor South	200	700	20,000	30	600,000	1.5	140,000	2.60
DuPont Edge Moor Cherry Island Staging Area	400	300	150,000	NA	150,000	1.5	120,000	0.76

Results of the site-specific high-end simulations are presented in the Table 7. The results for EPA's high end run are also presented for comparison. For many of the simulations, dilution attenuation factors (DAFs) were significantly higher than those of EPA, and many were orders of magnitude higher. Putting these much higher DAFs into the risk assessment would result in no unacceptable human risks from groundwater exposures.

In addition, during modeling runs it was determined that there may be incompatibility in recharge term versus the aquifer flow system. In some cases, the recharge term will contribute much more water to the aquifer system than would be indicated by the specified gradient and hydraulic conductivity, resulting in dilution attenuation factors that may be underestimated by orders of magnitude. This will be discussed in the modeling report to be submitted later.

These site-specific simulations indicate that under the highly unlikely scenario that leachate would reach a hypothetical well (most leachate would either be captured by landfill leachate collection systems or migrate to the adjacent rivers), there would not be an unacceptable risk.

Table 7
Site Specific Groundwater Modeling Results

Site	Metal	Distance to Well (m)	Unsaturated Zone K_d (cc/g)	Vadose Zone Thickness (m)	Vadose Zone Saturated Hydraulic Conductivity (cm/s)	Vadose Zone Saturated Hydraulic Conductivity (m/y)	Infiltration and Recharge rate (m/y)	Concentration (mg/l)	Dilution Attenuation Factor
High-End	Manganese	427	113	5.79	7.17E-05	189	0.2007	2.41 E+00	6.8
High-End	Thallium	427	31.6	5.79	7.17E-05	189	0.2007	1.84E-03	6.5
1	Manganese	1600	113	9.14	1.00E-05	252	0.1641	1.73E-02	942.2
1	Manganese	1600	430	9.14	1.00E-05	252	0.1641	1.42E-08	1,147,887,323.9
1	Thallium	1600	31.6	9.14	1.00E-05	252	0.1641	8.55E-04	14.0
1	Thallium	1600	500	9.14	1.00E-05	252	0.1641	0.00E+00	No exposure
2	Manganese	1600	113	9.14	1.00E-05	252	0.1641	1.54E-04	105,844.2
2	Manganese	1600	430	9.14	1.00E-05	252	0.1641	2.23E-10	73,094,170,403.6
2	Thallium	1600	31.6	9.14	1.00E-05	252	0.1641	2.45E-04	49.0
2	Thallium	1600	500	9.14	1.00E-05	252	0.1641	0.00E+00	No exposure
3	Manganese	1600	113	9.14	1.00E-05	252	0.1641	1.75E-04	93,142.9
3	Manganese	1600	430	9.14	1.00E-05	252	0.1641	4.62E-10	35,281,385,281.4
3	Thallium	1600	31.6	9.14	1.00E-05	252	0.1641	2.70E-04	44.4
3	Thallium	1600	500	9.14	1.00E-05	252	0.1641	0.00E+00	No exposure
	Site 1 - DWSA Cherry Island berms/cover								
	Site 2 - Port of Wilmington South Harbor								
	Site 3 - Dupont Edge Moor Cher Island Stain Area								

Response 22-7g: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule. We are responding to this comment because it raises issues relevant to our risk assessment for thallium. EPA appreciates the commenter identifying the discrepancies between the risk assessment background document and the model input data files. These discrepancies represent documentation errors in the background document rather than errors in the modeling analysis.

For the reasons stated previously, EPA disagrees with aspects of the commenter's characterization of the hydrogeology of the industrial corridor area. EPA also finds the hydraulic conductivity value selected to represent the Columbia Aquifer (i.e., 252 meters per year) to be unusually low and seems inconsistent with the grain size information submitted subsequently.

The K-value determined by a slug test in well PZ-12 screened in the Columbia Aquifer is representative of a silty sand aquifer but may be too low compared to published values for this aquifer. Published data (Johnston, 1977) for the Columbia Aquifer in central and southern Delaware, ranging between 550 and 2,700 meters per year, suggest that the vertical K-value may be higher. However, it is noted that the thickness and texture of the Columbia Aquifer in New Castle county may not be comparable to the aquifer in Kent County given that the depositional histories are not the same. The logs provided do indicate that the texture in the corridor is a medium to coarse slightly silty sand occasionally with fine gravel which would suggest a higher K-value than that for a fine or silty sand texture. (EPA notes that additional information submitted by the commenter indicates considerably higher hydraulic conductivities, confirming EPA's concerns. According to information provided by the commenter, hydraulic conductivities derived from pump tests for the Columbia range from 1,000 to 20,000 meters per year.)

More fundamentally, EPA believes that off-site disposal is a potential disposal option for the waste and, therefore, a site-specific modeling analysis is inappropriate for assessing the risks from waste disposal. In addition, DuPont officials have indicated the company no longer plans to market the material for use in the industrial corridor. Therefore, the commenter's focus on the corridor appears misplaced.

EPA has examined the incompatibility issue raised by the commenter and elaborated upon in subsequent materials furnished by the commenter. EPA acknowledges the potential for incompatibilities to exist between values specified for infiltration and recharge and the hydraulic properties of the aquifer. However, EPA disputes the impact these conditions have on the distribution of modeled dilution and attenuation factors (DAF's). When combinations of recharge/infiltration rates and aquifer characteristics are screened out that cause model-generated mounding to exceed the depth of the unsaturated zone (i.e., rise above the elevation of the ground surface), the resulting distribution of DAF's is similar to the distribution generated for the proposal. Significantly, it is similar for combinations at the lower percentiles (i.e., the portion of the distribution associated with low DAF's and, therefore, high risks). EPA concludes that the magnitude of the differences do not change the overarching conclusions regarding the potential risks posed by thallium in Iron Rich.

Comment 22-8: EPA Should Fully Explore Conditional, Concentration-based Listings and Other Alternatives (Including an Enforcement Agreement Approach) Before Moving Forward on Any K178 Listing for Solids from the Production of Titanium Dioxide

EPA has previously acknowledged that generic listings of hazardous wastes often are not required to assure environmental protection, because many wastes are not hazardous when managed in particular ways. In the recently promulgated Chlorinated Aliphatics listing rule, EPA reiterated this point when issuing a conditional listing for EDC/VCM wastewater treatment sludges. EPA listed these sludges as hazardous waste only when managed in ways different from those EPA found to be protective. 65 FR 67068, 67070/2-3 (Nov. 8, 2000). As the Agency stated:

> It does not make sense to list the bulk of EDC/VCM wastewater treatment sludges managed safely in landfills based upon the management approaches used by two facilities . ....A contingent management listing approach is within EPA's statutory authority . . . . EPA concludes that where a waste might only under limited management scenarios, ... the Agency is not required to list a waste as hazardous.

65 FR 67096/1-2.

Similarly, the Agency has recognized the benefits of moving toward a performance-based listing system and has proposed to incorporate performance standards directly into the listing descriptions using concentration-based listing criteria. For example, in the Dye and Pigment proposed listing rule, EPA recognized that concentration-based listings would assure protection and would avoid unnecessary and improper listing of wastes that are not in fact hazardous.

> A waste would be hazardous unless a determination is made that it does not contain any of the constituents of concern at or above specified levels of concern. This approach draws from the concept of the characteristic approach to defining a hazardous waste, in that whether a waste is hazardous depends on the levels of key constituents in the waste.

64 FR 40191 (Jul. 23, 1999).

Finally, the Agency has also considered even more innovative alternatives to traditional listings. In the Dye and Pigment proposed listing rule, the Agency discussed the possibility of employing enforceable agreements between industry and EPA that would restrict the management of wastes to practices the agency has determined do not pose significant risks. 59 FR 66072 (Dec. 22, 1994). Thus, the enforceable agreement approach provides benefits similar to a conditional listing approach.

EPA has not presented any conditional, concentration-based, or other alternatives, such as enforceable agreements, for consideration in the Inorganic Chemical Manufacturing Identification and Listing proposal. Yet, DuPont believes that the production of titanium dioxide presents unique opportunities for the consideration of each of these alternatives.

DuPont, therefore, urges the Agency to consider alternative approaches to the generic listing described in the proposal, such as those EPA has proposed and successfully implemented in a number of recent hazardous waste listing determinations (e.g., wood preserving rule, carbamate listings, chlorinated aliphatics, dyes and pigments proposed rule). These approaches have all recognized that many of the wastes generated by industry are hazardous only when managed in particular ways. These alternative approaches allow the regulated community to modify their manufacturing processes or waste management practices to avoid generating hazardous wastes in the first instance, thereby avoiding unnecessary regulation and the public and private costs of regulatory oversight, without compromising protection of human health and the environment. In particular, these alternatives avoid the perverse effects of the RCRA's mixture and derived-from rules for treatment residues and new treatability groups no longer containing the regulated constituents of concern or demonstrating treatment to concentrations, or with technologies, deemed acceptable to allow regulation outside of Subtitle C of RCRA.

DuPont believes the Agency should not allow this proposal to move forward without consideration of these important alternatives to a K178 listing. As DuPont's comments indicate, DuPont is requesting major revisions to the proposal. Against this backdrop, EPA should refine the scope of any final listing by providing conditional listing, concentration-based listing, or enforceable agreement alternatives.

Comment 22-8a: The Agency Should Consider a Conditional Listing Approach

In *Military Toxics Project v. EPA,* 146 F.3d 948 (D.C. Cir. 1998), the court found that, if EPA concludes that a waste might pose a hazard only under limited management scenarios, EPA can reasonably and permissibly determine that the waste should be regulated as hazardous only under those scenarios. In the *Military Toxics Project* case, EPA reasonably determined that waste munitions would not pose a hazard if managed in accordance with existing military munitions handling regulations.

Similarly, in the Chlorinated Aliphatics final rule, the Agency finalized it’s proposed "contingent management listing" for EDC/VCM wastewater treatment sludges based upon the Agency's determination that these sludges only warrant listing when managed in land treatment units, but not when managed in landfills. 65 FR 67068 (Nov. 8, 2000). In doing so, the Agency appropriately concluded that "it does not make sense to apply a traditional listing approach *(i.e.,* list all wastes regardless of management practice) based upon a practice occurring at one facility, especially if a more tailored listing can prevent this risk". 65 FR 67097 (Nov. 8, 2000).

In contrast, in the proposed rule, EPA has developed it's proposed listing for K178 (solids from the production of titanium dioxide) based upon current practices at one facility (modeling disposal in an off-site, unlined industrial landfill), knowing full well that two of the three chloride-ilmenite facilities exclusively dispose of similar wastes in dedicated on-site landfills subject to state industrial non-hazardous waste regulations. [Note: Chloride- and sulfate- process also presently discard similar waste in on-site landfills]. This proposed traditional listing approach appears to ignore several important factors. For example,

- The Agency modeled the on-site landfill at the Tennessee facility for potential releases to surface water and determined that "concentrations of the constituents of concern in the river are likely to be well below the national AWQC for human health and aquatic life for these constituents". 65 FR 55762 (Sept. 14, 2000). Yet, despite exclusive disposal of the Tennessee facilities wastes in a dedicated on-site landfill subject to state industrial non-hazardous waste regulations, EPA has still proposed to list these same wastes based upon disposal in an off-site, unlined industrial landfill.

- For waste concentration inputs to the risk modeling, EPA relied exclusively on data obtained from samples EPA collected from the Delaware facility. As shown in Table III-54 in the proposed rule, similar wastes generated at DuPont's Tennessee and Mississippi facilities are significantly different. 65 FR 55761 (Sept. 14, 2000).

- In completing its risk assessment, EPA relied upon its "usual distance-to-well assumptions for off-site landfill, and assumed hydrogeologic conditions that are representative of the principal soil and aquifer types present regionally (within a 100 mile radius) of the [Delaware] facility". 65 FR 55762 (Sept. 14, 2000). This is inappropriate. The comparative plausible waste management scenario for DuPont's Tennessee and Mississippi facilities is disposal in on-site landfill. Clearly the hydrogeologic conditions within a 100 mile radius of the Delaware facility do not accurately reflect the hydrogeologic conditions at either of DuPont's Tennessee or Mississippi facilities.

  Furthermore, DuPont notes that the Agency assessed the management practices at each site individually using site-specific factors in its evaluation of "wastewater treatment sludges from commingled chloride- and sulfate- process wastewaters". 65 FR 55758 (Sept. 14, 2000).

- The Mississippi facility "provided detailed information regarding the amounts of solids present in each of the wastewaters managed in [its] wastewater system, demonstrating that there is a small contribution (approximately 3%) of non-exempt solids (i.e., solids from oxidation and finishing) in the wastewater treatment sludge". 65 FR 55761 (Sept. 14, 2000). Based upon this relatively small contribution of nonexempt solids to the volume of exempt solids, EPA should have chosen, consistent with its evaluation of three Chloride process facilities, "not to attribute any risks to the non-

> exempt portions of these commingled solids". 65 FR 55753 (Sept. 14, 2000). Instead, EPA suggests that the "sampling and modeling of the ...Delaware [site] is an appropriate surrogate" for the Mississippi facility. 65 FR 55761 (Sept. 14, 2000). However, "the percentages of non-exempt solids in the commingled wastes at the Tennessee and Delaware sites are higher than at the Mississippi site". 65 FR 55761 (Sept. 14, 2000). [DuPont's notes a discrepancy with respect to the percent non-exempt solids EPA reports as present in wastewater sludge at its Tennessee facility. At 65 FR 55761 EPA states that it has "determined that the wastewater sludge at contains approximately 74% non-exempt solids", whereas Table 3.24 in the Titanium Dioxide Listing Background Document reports that the same sludge contains approximately 6.2% non-exempt solids]. Consistent with the Agency's determination for the three Chloride process facilities, DuPont believes that whenever the contribution of non-exempt solids to the volume of exempt solids is small, that EPA should choose "not to attribute any risks to the non-exempt portions of these commingled solids". 65 FR 55753 (Sept. 14, 2000).

Should the Agency decide to move forward with the proposed listing, DuPont believes that, consistent with the *Military Toxics Project* case and Chlorinated Aliphatics final rule, the Agency should fully evaluate a conditional listing approach for the Titanium Dioxide sector. For example, DuPont believes that allowing solid residuals from the production of titanium dioxide to continue to be managed in on-site units achieves necessary protection of human health and the environment. Further DuPont also believes that such materials managed off-site in 40 CFR 258 municipal solid waste landfills, should be considered protective. Thus, DuPont strongly encourages the Agency to acknowledge through promulgation of a conditional listing that little additional benefit would be gained by requiring that all solid residuals from the production of titanium dioxide be managed in accordance with Subtitle C of RCRA.

Response 22-8a: As noted in earlier responses, the final listing is limited to nonexempt ferric chloride solids. Neither the Tennessee or the Mississippi facilities generated this waste, thus the only plant that generates the listed waste is the Delaware facility. Therefore, the management practices at the Tennessee or Mississippi plants are not directly relevant to the potential risks from the listed solids. Furthermore, DuPont has recently indicated that it no longer intends to market the Iron Rich^{TM} and that it is considering disposal in off-site landfills. Thus, our evaluation of the risks presented by the waste solids (using data for the waste at the Delaware site) in an industrial or municipal landfill is appropriate, and represents a reasonable approach to assessing risks for a listing determination. Our analyses show that disposal in off-site industrial or municipal landfills presents significant risk. We see no opportunity for promulgating a conditional listing, because our analysis indicates that the most plausible management practices for ferric chloride solids show significant risk.

Comment 22-8b: The Agency Should Consider A Concentration-Based Listings Approach

DuPont also submits that EPA should consider a concentration-based listing approach for K178. In the Dye and Pigment proposed listing rule, EPA cited three significant benefits of adopting concentration-based listing. These are: promoting source control and waste minimization; responding to variability of processes and wastes; and reducing reliance on confidential business information. All of these benefits are applicable to the Agency's proposal to list K178.

C [A] concentration-based listing approach may provide an incentive for hazardous waste generating facilities to modify their manufacturing processes or treat their wastes. For example, if a facility has a listed hazardous waste based on constituent-specific concentration levels established by EPA, it also knows the required concentration levels of constituents in its waste below which its waste would become non-hazardous. Therefore, the facility may decide to modify its manufacturing process in order to generate a nonhazardous waste. Thus, this approach encourages waste minimization.

64 FR 40191 (July 23, 1999). A concentration-based listing for solid residuals thus would promote source control and waste minimization, encouraging facilities to generate wastes that are not hazardous. In contrast, the current proposal provides a broad, generic listing that provides little or no incentive for technological advancement and waste minimization.

C Batch operations may result in highly variable wastes at the same facility or different facilities. A concentration-based approach allows the variable wastes generated at these facilities to be evaluated individually for hazard, so only the truly hazardous wastes are listed. This tailored approach is more cost effective for the industry than a standard listing, and avoids the unnecessary regulation of nonhazardous waste.

64 FR 40191 (July 23, 1999). Although EPA's rationale here relies chiefly on the wide variety of products produced by dye and pigment manufacturers, these manufacturers normally produce these products from "manufactured feedstocks", whereas production of titanium dioxide relies upon "raw or non-manufactured feedstocks". The variability in these "raw or non-manufactured feedstocks", in addition to site-to-site variations in technology and operating practices, leads to variations in the volume and toxicity of the wastes produced, as evidenced by data presented in other sections of DuPont's comments.

C [M]any manufacturers in the dye and pigment industries want to keep facility-specific product and waste information confidential. These manufacturers are concerned that release of such information could cause competitive harm. A concentration-based listing allows [EPA] to rely less on CBI, since [EPA does] not use this information directly to set the listing concentrations. This means we don't use specific information, such as product formulations or concentrations of constituents in the wastes, to set hazardous concentration levels for constituents of concern.

64 FR 40191 (July 23, 1999). Maintaining confidential business information is an important concern for DuPont.

Another significant benefit of a concentration-based listing approach is relief from the perverse effects of RCRA's mixture and derived-from rules. The Agency's failure to propose a concentration-based listing for solid residuals from the production of titanium dioxide conflicts with EPA's concurrent activities to provide meaningful relief from the overbreadth of these very same rules (i.e., Hazardous Waste Identification Rule; 64 FR 63382 (Nov. 19, 1999)). Thus, without a concentration-based listing, the only relief from these rules requires a formal rulemaking process (i.e., delisting), unnecessarily subjecting DuPont and the overseeing agency to all the delays and costs attendant to this burdensome process. DuPont strongly encourages the Agency to consider this approach.

<u>Response 22-8b:</u> As noted by the commenter, we have proposed (but not yet finalized) concentration-based listing in some cases. However, we do not believe that the K178 would be an appropriate candidate for such a listing for several reasons. First, ferric chloride solids are currently generated at one site. The available data indicate that this waste presents potential risks as it is now generated. Therefore, there seems little reason to design a concentration-based listing for such a case. Furthermore, we have considered concentration-based listings when batch processes are used by various manufacturers to make a variety of end products. In the case of K78, there is only one generator and the process is designed to make essentially one product, titanium dioxide. The variety of data collected by DuPont and EPA for this listing determination appears to show that thallium is consistently present at levels of concern. In addition, we have concerns about the potential risks from the dioxin and furans present in this material (although we have not assessed these risks quantitatively). Thus, we would not expect the waste to vary widely in constituent concentration at this one site. Given these facts, we see no compelling reason to attempt to design a concentration-based listing for K178.

<u>Comment 22-8c:</u> The Agency Should Consider an Enforceable Agreement Approach

Finally, DuPont encourages the Agency to consider an "enforceable agreement" approach, which would limit the type of management and locations for managing solid residuals to those that do not pose significant risks. This would wholly avoid the need to list any solid residuals from the production of titanium dioxide as K178 hazardous waste.

In the December 22, 1994 Dye and Pigment proposed rule, EPA requested comment on innovative ways of conducting listing determinations that could assure environmental protection with less cost than full regulation as a hazardous waste. One approach on which the Agency sought comment was:

> whether enforceable agreements between EPA and industry that restrict the use of certain waste management practices could affect the Agency's plausible mismanagement analysis and, in turn, affect the Agency's listing determination. EPA

> [specifically sought] comment on whether EPA should decide not to list such wastes (or retain a no list decision) if the agreements ensure that the wastes will not be managed in a manner that poses an unreasonable risk. A decision not to list based on such enforceable agreements could be based on the view that management practices that are prohibited in an enforceable agreement are not "plausible" because facilities within an industry covered by an enforceable agreement are unlikely to violate that agreement; i.e., use a risky management practice, especially if the agreement were to contain certain monetary or other sanctions for a breach or violation. Waste management practices that are not plausible because they are prohibited by such an agreement arguably need not be considered by the Agency in determining whether the waste poses "a substantial present or potential hazard ... when improperly treated, stored, transported or otherwise managed ...."

59 FR 66072 (Dec. 22, 1994).

Given the Agency's proposal to list only "nonwastewaters from the production of titanium dioxide by the chloride-ilmenite process" and the fact that the proposed listing is limited to three DuPont facilities, an enforceable agreement alternative to listing these solid residuals as hazardous waste is both a practical and logical alternative to a generic listing.

For example, subject to, successful negotiation of the specific terms of an enforceable agreement, DuPont would be willing to enter into an enforceable agreement approach that allows:

1. Disposal of "nonwastewaters from the production of titanium dioxide by the chloride-ilmenite process" in landfills subject to state industrial non-hazardous waste regulations.

2. Land-based beneficial use/re-use of waste derived products conditioned on results of site-specific groundwater pathway risk assessments. Such an approach may be self implementing and effective only upon receipt of written confirmation of receipt of a notification package by the overseeing agency.

3. Beneficial use/re-use of waste derived products conditioned on receipt of approval by the appropriate overseeing agency(s) in accordance with established state or local regulations.

DuPont encourages the Agency to consider this. DuPont will provide any reasonable assistance that the Agency may require to further evaluate this alternative and to enter into the requisite agreement.

Response 22-8c: As discussed earlier, DuPont has stated that the company would not pursue beneficial use options, because of the potential risks that could arise from dioxin contaminants in the material as it is currently formulated. DuPont stated that it is evaluating effective treatment processes to reduce the concentrations of organics in the material, and confirmed that it may well have to dispose of the material in an off-site landfill located outside of the corridor near the plant. Thus, there seems little reason to consider an enforceable agreement when these practices are no longer under consideration. Furthermore, relying on an enforceable agreement under RCRA presents a number of complex legal and policy issues.

Comment 22-9: EPA Has Inappropriately Established Land Disposal Restriction Treatment Standards for Manganese, Thallium, OCDD and OCDF and Has Erroneously Assumed Combustion Facilities Are Available to Manage These Solid Streams

Comment 22-9a: Land disposal restrictions (LDRs) standards are not appropriate for constituents that are not toxic or hazardous constituents

As noted by EPA at 65 FR 55767, land disposal restrictions consistent with the requirements of 3004(m)(1) are required by the RCRA statute as amended in 1984 for wastes which become hazardous due to listing determinations and that those treatment standards must of necessity prescribe levels or methods of treatment which substantially diminish the toxicity of the waste or substantially reduce the likelihood of migration of hazardous constituents. The requirement to set LDRs for hazardous wastes can reasonably be construed to limit the constituents for which standards may be set to those which are toxic and therefore pose a risk.

For example, as manganese is shown elsewhere in these comments to not pose a risk, manganese seems to be an unsuitable candidate for an LDR treatment standard.

Response 22-9a: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-9b: Land disposal restrictions (LDRs) standards are not appropriate for constituents in wastes that are not at levels that pose risks.

Given that thallium is shown by the data presented elsewhere in these comments to not be present in appreciable levels in this waste matrix, imposition of an LDR standard for thallium is not consistent with the statute. Imposition of a standard which will consistently be met in a waste on an as-generated basis is not appropriate under the intent of 3004(m). The data presented in these comments confirms that no waste stream proposed for listing exceeds 0.84 mg/kg (and most are nondetects at a reporting limit of 0.5 mg/kg) and therefore no waste stream would exceed a TCLP value of 0.042 mg/L opposite the proposed LDR standard of 0.2 mg/L. The thallium levels in wastewaters from the Delaware and Tennessee site presented at 65 FR 55764 in Table III-58 (<0.005 and .013 mg/L respectively) are more representative of the thallium concentrations which are likely to result from TCLP analysis of the solids proposed for listing as K178 than what the erroneous thallium results discussed elsewhere in these comments suggest. The Agency's own assessment for the proposal found no basis for listing the wastewaters and therefore no need for an LDR standard for such waste. There is similarly no basis for listing nor applying an LDR to the solids from the chloride-ilmenite process based on thallium content.

Response 22-9b: EPA disagrees with the commenter. EPA's record sampling showed thallium concentrations in the Iron Rich™ sample of 0.28 mg/L TCLP (65 FR 55761, September 14, 2000). Even if the record sampling event produced higher than normal concentrations of thallium (as the commenter claims), a treatment standard for thallium will ensure that potential risks from the constituent are minimized. In instances when the waste exhibits thallium concentrations below the treatment standard, no additional treatment will be necessary prior to land disposal.

Comment 22-9c: The proposed manganese treatment standard of solidification is inappropriate due to dissimilar waste matrice transfer

At 65 FR 55768, the Agency proposes solidification technology transfer from dilute low concentration manganese nonwastewaters to the solids proposed for listing as K178 while acknowledging that their own database includes no demonstrated technologies for handling wastes with the manganese concentrations anticipated in these wastes. As the Agency is well aware, transfer of treatability data from one waste matrix to another waste matrix creates treatability problems even when the concentration of contaminants are similar. To take the additional step of transfer from dilute waste matrices to concentrated ones seems unjustified in this case. The solids in question here are highly metallic in nature (principally containing non-toxic metals such as iron and aluminum) and the potential interference and competition for the binding agents typically employed in solidification processes are obvious in this type of waste matrix. Solidification is typically most effective in solids which are largely composed of inert materials with trace metals present. Therefore, presumption that treatment of these wastes by solidification would yield similar results to those presented at 65 FR 55768 even allowing for the 2.8 variability factor for mechanical limitations of treatment equipment is questionable. The variability factor does not address the more important question of waste matrix interference and its impact on solidification.

Response 22-9c: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today's final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-9d: The proposed thallium treatment standard for K178 is inappropriate since the wastes do not contain thallium

EPA identifies a treatment standard for thallium at 65 FR 55767. As discussed in sections

4.2 and 5.3, DuPont believes that the TCLP and SPLP thallium levels detected in these solids are laboratory contamination artifacts. DuPont analyses of the ores used in the manufacturing process for various ores used in the past year indicate that thallium is generally present at levels below 50 ug/kg. The highest level we have identified in any ore is 171 ug/kg, which would equate based on solids/ore ratio to a theoretical maximum of 285 ug/kg on a total composition basis for any solid created from the process. This equates to a maximum theoretical TCLP value of 14.3 ug/L for any solid generated, opposite a proposed LDR standard of 200 ug/L. Based on the apparent absence of leachable thallium and the low level of thallium on a total composition basis in the ingredient ore, DuPont submits that the EPA has not justified the conclusions presented in Table III-56 at 65 FR 55762.

Because the Agency was relying on questionable data, inclusion of thallium as a basis for listing of K178 under 40 CFR 261 Appendix VII is inappropriate under the Agency criteria delineated at 40 CFR 261.11(3). By extension, imposition of a land disposal restriction on thallium for this waste is not appropriate. The basis for DuPont's assertion that thallium is not present is based on the sampling program conducted in response to this proposed rulemaking, including not only an assessment as to the lack of thallium present in these wastes but also the lack of any appreciable thallium in the ores.

<u>Response 22-9d</u>: We believe the treatment standard for thallium in K178 wastes is appropriate. EPA’s record sampling showed thallium concentrations in this waste of 0.28 mg/L TCLP (65 FR 55761, September 14, 2000). As explained above in response 22-5a-1, we believe that our sample and analysis are valid. Moreover we do not believe that DuPont’s estimate of maximum TCLP values based on ore content is valid (see response 22-5a-4). Even if the record sampling event produced higher than normal concentrations of thallium (as the commenter claims), a treatment standard for thallium will ensure that potential risks from the constituent are minimized. In instances when the waste exhibits thallium concentrations below the treatment standard, no additional treatment will be necessary prior to land disposal.

Absent setting a thallium standard specifically in K178, the subject wastes would still need to be treated for thallium if it is reasonably expected to be present should the waste also exhibit the properties of a characteristic waste. (See 40 CFR 268.9(b).) Having found thallium in the EPA samples at levels above the UTS, we believe it is reasonable to expect it to be present.

<u>Comment 22-9e:</u> EPA believes there is adequate combustion capacity for the proposed inorganic wastes. This assumption is based on volumes of potential wastes that are not in line with actual generated volumes.

Under Section N, "Proposed Treatment Standards Under RCRA's Land Disposal Restrictions", 65 FR 55771, EPA proposes not to grant a capacity variance for nonwastewater forms of K178. This determination is based on 300,000 tons of available commercial combustion capacity for sludge and solids versus the calculated need for 7300 tons of needed capacity

(65 FR 55770) for this proposed new waste.

DuPont has shown in previous comments that the amount of waste potentially needing treatment by this rule could be as high as 167,000 tons because of the overreaching manner in which the proposed rule has been written. DuPont believes that based on potential volumes needing treatment versus available capacity that it will not be feasible to incinerate these proposed wastes.

It is DuPont's understanding that the only incinerator in the United States that will have permitted capacity for dioxin containing wastes is the Aragonite facility in Utah (permitting in progress). The maximum permitted capacity for bulk solids at this facility will be 16,200 pounds per hour (8 tons per hour). If the facility incinerated 8 tons an hour, 24 hours a day, 365 days a year, it could consume 70,000 tons per year. Thus there is not sufficient capacity in the United States to incinerate all of the waste DuPont would generate under this proposed rule.

<u>Response 22-9e:</u> The commenter provided few details that would explain the discrepancy between EPA's estimate and its own estimate, and therefore EPA does not necessarily agree with the commenter regarding this estimate.[18] Further, the finalized listing definition is narrower in scope than what was proposed, only one facility (rather than three) are expected to generate the waste, and the one facility may be able to segregate its waste to reduce its generated K178 quantity. However, even if EPA used this higher waste quantity in its capacity assessment, sufficient capacity would be available to treat generated K178 wastes.

EPA notes that DuPont's estimated volume of waste requiring incineration is still below the estimated 300,000 tons of available commercial combustion capacity for sludge and solids identified in the proposed rule. EPA disagrees with DuPont's claim that K178 can only be managed at one incinerator, with available commercial treatment capacity of only 70,000 tons. EPA notes that the land disposal restrictions for K178 are identical to those finalized for F032 (wood preserving wastes, 62 FR 26000, May 12, 1997) and K174 (chlorinated aliphatics wastes, 65 FR 67110, November 8, 2000). These treatment standards (as well as the treatment standards proposed for K178) can be met by the technology-specific standard of CMBST, defined as '(1) combustion units operating under 40 CFR 266, (2) combustion units permitted under 40 CFR part 264, subpart O, or (3) combustion units operating under 40 CFR 265, subpart O, which have

---

[18] As correctly noted by the commenter, EPA estimated in the proposed rule that approximately 7,300 tons per year of K178 waste would require alternative treatment capacity. This estimate was based on non-confidential information sources, since estimates provided by K178 generators in the RCRA 3007 surveys (described in the proposed rule) were claimed as RCRA confidential business information. However, while EPA welcomes the additional information provided by the commenter in reference to K178 generation quantities, EPA found only qualitative references in DuPont's comment concerning how this estimate was derived. Once such difference includes EPA's assumption that only 10% of the chloride solids will be Bevill nonexempt, while DuPont claims that all of its generated chloride-ilmenite solids will have to be treated and disposed of as K178 wastes.

obtained a determination of equivalent treatment under 268.42(b). Additionally, EPA verified through telephone conversations that several facilities can, in fact, accept wastes with such a treatment standard (this information is presented in the Capacity Background Document).

Comment 22-9f: DuPont believes a capacity variance will be essential if the proposed rule is promulgated as written.

The Agency has proposed that the land disposal restriction treatment standards for the affected wastes covered under the rulemaking would become effective when the listing determinations become effective per 3004(h)(1) presuming that there is sufficient protective treatment capacity for the waste available. The nature of this waste and the likely resistance of available incineration capacity to accept this waste for combustion lead DuPont to the conclusion that a capacity variance will be necessary if the listing is finalized as proposed and if the land disposal restriction treatment standard remains based on combustion.

Other incineration and disposal permitting issues for mining wastes, as were recently raised in FMC's Case-By-Case extension to Land Disposal Restrictions (65 FR 12233, March 8, 2000), will also make it difficult to impossible to comply with the proposed treatment standards.

DuPont believes a capacity variance will be essential if the proposed rule is promulgated as written.

Response 22-9f: For the reasons described in its response to Comment 22-9e, EPA is not granting a national capacity variance for K178. EPA does not agree that insufficient treatment capacity exists, and the commenter has not presented information supporting its claim that commercial facilities would resist accepting this waste for combustion. EPA also disagrees that the issues identified in the proposal to extend the LDR effective date for FMC are necessarily relevant in this case. In the FMC proposal, EPA identified that the presence of elemental phosphorous, the potential for generating phosphine gas, the presence of Naturally Occurring Radioactive Material (NORM), and the volume of wastes to be managed resulted in FMC's finding that no commercial facilities would accept these wastes for treatment (65 FR 12239, March 8, 2000). In contrast, neither EPA nor the commenter has identified such concerns with regard to K178.

Comment 22-9g: EPA should delete proposed OCDD and OCDF treatment standards for K178 listing.

EPA has proposed to set OCDD and OCDF treatment standards for the K178 listing (65 FR 55767).

Response 22–9g: See our response to the more specific Comments 22-9g-1 through 22-9g-4 below.

Comment 22-9g-1: WHO-TEFs, endorsed by EPA in this proposal, demonstrate that OCDD and OCDF do not drive toxicity of dioxin/furan contamination that may be present in proposed K178 wastes.

Since the toxic equivalence factors (TEF) used by WHO and EPA for OCDD and OCDF are each 0.0001, it appears that EPA agrees that neither compound drives dioxin risk.

<u>Response 22–9g-1</u>: We first wish to clarify that potential risks from dioxin congeners were not evaluated during the risk assessment for K178 and were not proposed as a basis for listing. However, we are promulgating treatment standards for dioxin and furan congeners in K178, because toxic dioxin and furan congeners are present in this waste at concentrations well above the treatment standards. For example, OCDF was measured in EPA record samples at 58 ug/kg and 202 ug/kg, well above its treatment standard of 5 ug/kg (see Tables 2-9 and 2-10, EPA's "Best Demonstrated Available Technology (BDAT) Background Document for Inorganic Chemical Production Wastes - K176, K177, K178" (for the proposed rule)). Therefore, in this particular case, the presence of elevated concentrations of OCDF influenced our decision to promulgate treatment standards even though its TEF was relatively low.

Secondly, applying the TEF cited by the commenter[19] to the 2,3,7,8-TCDD cancer slope factor used in the risk assessment for the chlorinated aliphatics rulemaking (see, e.g., EPA's "Risk Assessment, Technical Background Document for the Chlorinated Aliphatics Listing Determination" (Addendum) September 29, 2000) still demonstrates significant toxicity for these congeners. Applying these data, the slope factor for OCDD and OCDF is effectively 15.6 $(mg/kg\text{-}day)^{-1}$. This is by comparison ten times the slope factor for arsenic, an Appendix VIII constituent and known carcinogen.[20]

Finally, OCDD may undergo photolysis, resulting in the formation of more toxic congeners.[21] For these reasons, EPA concludes that adopting specific treatment standards (i.e., numerical standard or a technology of CMBST) for these congeners is warranted for K178 wastes.

<u>Comment 22-9g-2:</u> OCDD and OCDF are not toxic.

A long-term study was reported that dramatically demonstrates that dioxin-like effects are not produced by OCDD in animals-even at high dose levels of OCDD (Wermelinger, et al. 1990).

---

[19] The toxicity equivalency factor (TEF) of 0.0001 for OCDD is developed by the World Health Organization.

[20] The cancer slope factor for arsenic is 1.5 $(mg/kg\text{-}day)^{-1}$, as identified in EPA's Integrated Risk Information System (IRIS). A higher slope factor indicates more potent carcinogenic effects.

[21] Miller, Glenn; Hebert, Vincent; Miille, Michael; Mitzel, Robert. "Photolysis of Octachlorodibenzo-p-dioxin on Soils: Production of 2,3,7,8-TCDD." Chemosphere. Volume 18, pp. 1265-1274 (1989).

An important structural difference exists between OCDD and OCDF and the other 2,3,7,8-PCDD/F compounds that have been shown to exhibit dioxin-like toxicity. OCDD and OCDF are the only 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) congeners that contain chlorine atoms at each nonlateral position of the dibenzo-p-dioxin and furan ring structure (Koester, et al. 1988). Chlorination at each nonlateral position of the dibenzo-p-dioxin and furan ring structure results in a number of significant structural changes that adversely impact the ability of OCDD and OCDF to efficiently bind to TCDD-specific enzyme binding sites (Mason, et al 1986).

OCDD has not shown that it produces the same toxicological profile as PCDDs and PCDFs (Schwetz, et al. 1973). The exception is a study reporting that repeated exposure to OCDD results in dioxin-like effects (Couture 1988). A close review of the study, however, shows that repeated exposure to OCDD results only in slight and transient effects. In fact, for each parameter reported in the study, the initial elevation observed over the controls disappeared by the end of the study. Other investigators have not characterized the Couture study as demonstrating dioxin-like effects. In a report by Wermelinger, et al., the authors noted that "except for some lipid droplets in the liver, Couture et al. observed no other toxic effects as usually seen with the toxic PCDD/F." In the Wermelinger, et al. study, even after nine months (26 weeks longer than the Couture, et al. study) of OCDD treatment at high dose levels, no significant treatment related liver damage could be observed in the test animals. In support of this finding, OCDD produced no effects when evaluated by the National Toxicity Program in a two-year study.

For OCDF, essentially no toxicological data has been published in literature and, therefore, insufficient evidence exists to support the listing of this compound as an Appendix VIII hazardous constituent.

Response 22–9g-2: EPA disagrees with the commenter for several reasons.[22] First, there are data from subchronic studies for both OCDD and OCDF which demonstrate dioxin-like effects (Couture et al., 1988[23]; DeVito et al., 1997[24]). Couture et al (1988) is one of the best studies of OCDD and describes not only the effects but the importance of study design in examining the effects of OCDD. Couture et al. (1988) demonstrate toxic response of OCDD following

---

[22] EPA first identified these data in its final rule for listing chlorinated aliphatics wastes, which finalized EPA's action to include OCDD and OCDF on 40 CFR 261 Appendix VIII and to add them to the list of universal treatment standards at 40 CFR 268.48.

[23] Couture, L.A., M.R. Elwell, and L.S. Birnbaum. "*Dioxin-like Effects Observed in Male Rats Following Exposure to Octachlorodibenzo-p-dioxin (OCDD) during a 13-week Study.*" Toxicology and Applied Pharmacology, Vol. 93, Pp 31–46, 1988.

[24] Michael J DeVito, et al. "Dose-Response Relationships for Polyhalogenated Dioxins and Dibenzofurans Following Subchronic Treatment in Mice. I. CYP1A2 Enzyme Activity in Liver, Lung, and Skin." *Toxicology and Applied Pharmacology*. Dec 1997 v 147 n 2 267.

subchronic exposures. In addition, this study also provides tissue concentrations at which these effects are observed. Couture et al. (1988) demonstrate that the absorption of OCDD is dependent upon both dosing volume and concentration of the solution. The higher the concentration the lower the absorption, while the larger the volume (up to 5 ml/kg) the greater the absorption. Hence, high dose single exposures are unlikely to induce significant effects due to the limited absorption of OCDD. In contrast, low dose repeated exposures will allow for the bioaccumulation of OCDD, which eventually leads to biological effects. This is clearly demonstrated in the Couture et al study (1988). The repeated exposure to 1 ug/kg of OCDD in a dose volume of 5 ml/kg produces time dependent effects which are also associated with increasing tissue accumulation of OCDD. OCDD induces hepatic CYP1A1 activity and protein induction of CYP1A1 occurred as early as two weeks after treatment, and this response increased with time and with hepatic OCDD accumulation. Induction of CYP1A1 is a dioxin-like effect and is indicative of activation of the Ah receptor. Hepatic cytoplasmic vacuolization in the livers was also induced in a time dependent manner, first occurring after 40 doses and increasing incidence and severity was reported after 65 doses of OCDD.

EPA disagrees with the commenter’s argument that these effects are transitory or of uncertain toxicological significance. First, the cytoplasmic vacuolization in the liver increased in incidence and severity in a time dependent manner. The increase incidence and severity of these lesions was associated with increasing hepatic concentrations of OCDD. Animals at the last time point examined in the study of Couture et al (1988) demonstrated the highest incidence and severity of these lesions; it is difficult to describe them as “transitory” as the commenter suggests, given that these effects worsened over the last five weeks of the study. Indeed, hepatotoxicity can be considered as part of a continuum of events leading to necrosis or carcinogenicity. Demonstration of events early in this continuum, such as cytoplasmic vacuolization, are cause for concern. Indeed, dioxins are endocrine disruptors and hormonal imbalances are expected to be induced by OCDD and other dioxins. These hormonal imbalances should be considered adverse responses based on our understanding of the endocrine disrupting actions of these chemicals.

EPA also notes that not only was enzyme activity induced by OCDD in the rats, but CYP1A1 and CYP1A2 protein were also increased as demonstrated by western blot analysis (Couture et al., 1988). These proteins have been implicated in playing important roles in oxidative damage and porphyria (Sinclair et al., 2000).[25] According to Nebert and colleagues, “metabolism of endogenous and exogenous substrates by perhaps every P450 enzyme, but certainly CYP1A1 and CYP1A2 (which are located, in part, in the mitochondrion), have been shown to cause reactive oxygenated metabolite (ROM)-mediated oxidative stress” (Nebert et al., 2000).[26] Ames and

---

[25] Sinclair, PR; et al. CYP1A2 is essential in murine uroporphyria hexachlorobenzene and iron. Toxicol Appl Pharmacol 2000 Jan 1; 162(1):60–7.

[26] Nerbert, DW; et al. Role of the aromatic hydrocarbon receptor in the oxidative stress response, cell. Biochem Pharmacol 2000 Jan 1; 59(1):65–85.

colleagues have clearly demonstrated the role of CYP1A1 in oxidative stress (Park et al., 1996).[27]

The commenter cites a number of studies suggesting that OCDD is not toxic, in contrast to Couture et al. (1988). The studies cited are generally inadequately designed to address the toxicity of OCDD. Several studies have demonstrated that, while OCDD is poorly absorbed in biological systems (Norback et al, Birnbaum and Couture, 1988[28]; Couture et al., 1988), it can bioaccumulate through repeated exposures to low concentrations. In addition, in the Couture et al. study, it took at least 40 doses over approximately nine weeks before enough of the chemical could accumulate to produce alterations in liver histology. Acute, single exposures to high concentrations of OCDD are unlikely to result in significant accumulation to induce a toxic response since very little of the dose shall be absorbed. In fact that is one of the conclusions in the McConnell et al study (1978).[29] Hence the acute studies on the effects of OCDD demonstrated none of the typical signs of dioxin-like toxicity due to the limited absorption of the chemical. Other studies have to a lesser or greater degree attempted subchronic exposures. However, these studies either are too short (Holsapple et al (1986))[30,31] or use too concentrated a dosing solution (Norback et al., 1975).[32] In either case, too little OCDD was absorbed to induce effects.

The commenter cites a study by Wermelinger et al. (1990)[33] as evidence that OCDD does not induce dioxin-like effects. EPA strongly disagrees with this conclusion. This manuscript was published as an extended abstract from the dioxin meetings (Organohalogens 1:221-224). These data clearly demonstrate that both OCDD and OCDF administered in the diet result in clear dioxin-like activity. Both OCDD and OCDF resulted in dose-dependent increases in CYPiAi activity and decreases in thymic atrophy. These responses are clearly the hallmark of dioxin-like

---

[27] Park, JY; Shigenaga, MK; Ames, BN. (1996) Induction of cytochrome P4501A1 by 2,3,7,8-tetrachlorodibenzo-p-dioxin or indolo (3,2-b)carbazole is associated with oxidative DNA damage. Proc Natl Acad Sci USA 93:2322–2327.

[28] Birnbaum, LS; Couture, LA. (1988) Disposition of octachlorodibenzo-p-dioxin (OCDD) in male rats. Toxicol Appl Pharmacol 93:22–30.

[29] McConnell, EE; Moore, JA; Haseman, JK; et al. (1978) The comparative toxicity of chlorinated dibenzo-*p*-dioxins in mice and guinea pigs. Toxicol Appl Pharmacol 44:335–356.

[30] Holsapple, MP; Dooley, RK; McNerney, PJ; McCay, JA (1986) Direct suppression of antibody responses by chlorinated dibenzodioxins in cultured spleen cells from (C57B1/6 x C3H)F_1 and DBA/2 mice. Immunopharmacology 12:175–186.

[31] Holsapple, MP; McCay, JA; Barnes, DW (1986) Immunosuppression without liver induction by subchronic exposure to 2,7-dichlorodibenzo-p-dioxin in adult female B6C3F_1 mice. Toxicol Appl Pharmacol 83:445–455.

[32] D.H. Norback, et al., "Tissue Distribution and Excretion of Octachlorodibenzo-p-Dioxin in the Rat." 32 *Toxicol. Appl. Pharmacol.* 330-3 8 (1975).

[33] M. Wermelinger, H. Poiger, and C.H. Schlatter, "Results of a 9-Month Feeding Study with OCDD and OCDF in Rats" (1990), reprinted in 1 Organohalogen Compounds 221, 221 (O. Hutzinger and H. Fiedler (1991).

effects in experimental animals. The Wermelinger et al. study clearly supports the findings of Couture et al., that repeated low dose administration of OCDD results in dioxin-like effects. In addition, both Wermelinger et al. and Couture et al. provide similar estimates of the relative potency of OCDD, further supporting the inclusion of this chemical in the proposed treatment standards for K178.

The commenter cites a study by the National Toxicology Program in which a two year feeding study of OCDD produced no effects. We could not locate any reports of this study in the NTP databases. After contacting the NTP, it was determined that the study of OCDD was halted due to uncertain technical difficulties and no reports were ever prepared on any study of OCDD by the NTP.

The effects of OCDF are not as well studied as those of OCDD. Recent studies do demonstrate that subchronic exposure to OCDF demonstrate dioxin-like activities in mice (DeVito et al., 1997). The subchronic exposure resulted in EROD induction in liver, lung and skin (DeVito et al., 1997) and hepatic porphyrin accumulation (van Birgelen et al., 1996)[34,35] in these mice. These studies demonstrate that OCDF also possess dioxin-like activity.

We have concluded that OCDD and OCDF are hazardous constituents that contribute to the overall risks attributable to dioxin related compounds. Therefore, we find that treatment of these hazardous constituents are necessary to substantially diminish the toxicity of wastes that contain these constituents, and to substantially reduce the likelihood of migration of hazardous constituents from the waste so that short-term and long-term threats to human health and the environment are minimized.

Comment 22-9g-3: Since OCDD and OCDF are not toxic, EPA should not set treatment standards for OCDD or OCDF in the K178 listing.

In view of comments 9.7.1 and 9.7.2, EPA should not set treatment standards for OCDD or OCDF in the K178 listing pursuant to the comment in section 9.1.

[See original comment for references.]

Response 22–9g-3: EPA disagrees with the commenter. These compounds are currently included on the list of hazardous constituents in 40 CFR 261 Appendix VIII and have universal treatment standards as identified in 40 CFR 268.48. These constituents have been shown to have the

---

[34] van Birgelen, AP; Fase, KM; van der Kolk, J; et al. (1996) Synergistic effect of 2,2',4,5,5'-hexachlorobiphenyl and 2,3,7,8-tetrachlorodibenzo-p-dioxin on hepatic porphyrin levels in the rat. Environ Health Perspect 104(5):550–557.

[35] van Birgelen, APJM; DeVito, MJ; Akins, NJ; et al. (1996) Relative potencies of polychlorinated dibenzo-p-dioxins, debenzofurans and biphenyls derived from hepatic porphyrin accumulation in mice. Toxicol Appl Pharmacol 138:98–109.

potential to cause significant risks to human health or the environment (as shown by EPA’s response to Comments 22-9g-1 and 22-9g2 above) and their presence in K178 wastes should be mitigated to avoid such potential risks.

Comment 22-9g-4: EPA should defer extension of UTS for OCDD and OCDF to this listing until resolution of anticipated court challenges to EPA's promulgation of UTS per the Chlorinated Aliphatics final rule (Nov. 8, 2000).

Response 22–9g-4: EPA is promulgating treatment standards for dioxin congeners, including OCDD and OCDF, in K178 wastes as proposed, because treatment of these constituents is necessary to reduce the risks to human health or the environment that these constituents pose. The promulgated universal treatment standards for these congeners were not challenged in litigation stemming from the Chlorinated Aliphatics Final Rule.

Comment 22-9h: DuPont believes that if the final rule includes a treatment standard for manganese for K178 waste that the proposed level of 3.6 mg/L TCLP is inappropriate

According to the Tea Council (http://www.teahealth.co.uk/cgi gen/reslib/0000000325), leaf tea contains 350-900 mg/kg. If one assumes that all of the manganese in tea leaches under the toxicity characteristic leaching procedure (TCLP), the translated TCLP level range in that material, if a waste, would be 17.5 to 45 mg/L. The Agency should reevaluate the appropriateness of the proposed standard in light of the fact that a number of food items contain greater potential leachable levels of manganese than would be allowable under the hazardous waste program, where the potential to ingest is mitigated by dilution and attenuation.

Response 22–9h: We are deferring final action on all elements of our proposal that are specifically related to the waste constituent manganese. Please see section IV.B of the preamble to today’s final rule for a further discussion of our decision to defer final action on manganese-related elements of the proposed rule.

Comment 22-10: EPA Significantly Underestimates the Economic Impacts of this Proposed Listing

Based on the proposed regulations, as written, and the volumes of waste generated by DuPont, the actual estimated annual incremental treatment and disposal cost is as high as $216 million plus one time costs estimated in excess of $55 million for impoundment retrofitting and construction of hazardous waste storage areas, versus the total of $3 million estimated in the rulemaking for treatment and disposal. We believe that OMB needs to reevaluate the rule based on the fact that incremental costs to the chloride ilmenite titanium dioxide sector are severely underestimated.

Under Section VIII, Administrative Assessments, 65 FR 55775, the incremental cost for the treatment and disposal of newly listed wastes for chloride-ilmenite titanium dioxide sector is estimated at $2,937,000. The backup to this assertion can be found in the "Economic Analysis for Listing of Inorganic Chemicals, Notice of Proposed Rulemaking" published by Research Triangle Institute Center for Economics Research (referred to as ""Economic Analysis"" in these comments).

DuPont believes that the incremental costs for its three chloride-ilmenite plant sites have been severely underestimated by incorrect assumptions in the analysis. Using EPA's assumptions corrected for incorrect numbers and excluded costs (incineration) DuPont calculates the incremental cost of treatment and disposal to be $88 million (assumes the ability to segregate non-Bevill wastes) to $880 million annually (assumes segregation of non-Bevill wastes is not possible). Using 1998 actual volume numbers for the different streams, DuPont calculates the annual incremental cost of treatment and disposal to be $16 - 216 million. Additionally there is an estimated $55 million for retrofitting of surface impoundments and construction of hazardous waste storage facilities. Based on Executive Order 12866 we believe this regulatory action must be resubmitted to OMB for review of the realistic affect on the economy (65 FR 55774).

Our analysis indicates the following flaws:

Comment 22-10a: Using EPA's assumptions, corrected for excluded costs and incorrect numbers:

Cost of Incineration was not included.

The proposed treatment standards for K178 wastes require combustion as part of the treatment. The "Economic Analysis" did not include the cost of incineration in calculating the economic impact (though it was discussed and calculated in Appendix B of the "Economic Analysis"). Using the same assumptions from the "Economic Analysis" for volumes of wastes to be treated (page 4-11) and the quoted price for incineration (page B-4) results in an increase in the incremental cost of:

3 sites x 2200 mt/year x $727.51/mt = $4,801,566/yr additional annually

The cost of loading a truck was calculated assuming 1 truckload per quarter.

This is not physically possible so the number of truckloads must be factored in. EPA's rates for loading (page 4-4) and the assumption that a truckload consists of 20 tons yields the following calculation. The 4 truckloads subtracted out are those already accounted for in the existing calculation.

3 sites x [2200 mt/year/20 mt/load x ($103.00 - $61.80) - 4 x ($103.00 - $61.80)] = $13,102/yr additional annually

The combination of incineration coupled with stabilization/disposal will require a second truck loading and transportation cost to move the material from the incinerator to the stabilization/disposal location.

This loading and transportation is not offset by the nonhazardous case because this is a second handling. The "Economic Analysis" assumes 7% ash after combustion. Since these materials are essentially all inorganic we believe the total percent ash will be approximately 85%.

3 sites x 2200 mt/yr x ($103.00/20 + $239.38) x 0.85 = $1,371,813/yr additional annually

The "Economic Analysis" uses a 60% ilmenite ore (page 4-10) as the feedstock to the process. It is asserted (page 4-11, footnote 2) that the process will generate 4% chloride solids of which 10% will be Bevill nonexempt and thus require treatment.

The "Economic Analysis" document provides no explanation on how the 4% chloride solids number was arrived at. With a 60% titanium dioxide ore there is 40% that is not titanium dioxide. If one assumes all non-titanium dioxide would be waste solids then at a minimum, there will be 40% chloride solids to address. This section calculates the economic analysis assuming all non-titanium dioxide is present as solids. We recognize that due to the different methods used to handle the non-titanium streams at our sites that this overstates the actual solids that are generated, however, the 4% chosen by the Agency underestimates the solids generated. Section 10.4 of these comments calculates the economic impact based on actual solids numbers from our manufacturing facilities.

The assumption of 40% solids increases the amount of material to be treated from each site to rise from 2200 mt/yr to 22,000 mt/yr. With the addition of incineration, initial truck loading and the second loading and transportation costs, the incremental impact increases to:

3 sites x 22,000 mt/yr x[($239.38 - $79.00) + ($238.36 - $53.20 + $93.11) x 0.85 + $727.51 + ($103.00 - $61.80)/20 + ($103.00/20 + $239.38) x 0.85] = $88,065,780/yr (cumulative annual total)

Response 22-10a: EPA believes its estimate of costs and economic impacts for the proposed rulemaking was accurate based upon the information available to it at the time of proposal. EPA believes that DuPont's estimate of the cost of the rule does not reflect least cost behavior on the part of the firm. DuPont's cost estimate therefore results in an inflated estimate of the proposed rule's cost and economic impact to the company.

In undertaking our economic analysis, we assumed that DuPont would fully segregate its nonexempt nonwastewaters affected by the hazardous waste listing from the remainder of the titanium dioxide (chloride-ilmenite) production process so that the newly-listed wastes may be managed as hazardous waste without affecting the regulatory status of (or the costs associated with managing) the other, non-listed, materials. EPA believed at the time of proposal that DuPont would modify its production process to segregate its wastes into Bevill-exempt (i.e., not hazardous) wastes and nonexempt components prior to the rule's effective date. We therefore estimated compliance costs based on a relatively small volume of (segregated) nonexempt nonwastewaters. Modeling this smaller volume represents the fraction of the original material that would have remained subject to the hazardous waste listing after the effective date of the rule. We therefore did not include costs for incineration of any portion of the waste in our estimate of compliance costs. We believe that the commenter's statement that a second trip from an incinerator to a commercial treatment facility for stabilization is in error because many commercial incinerators have stabilization capacity onsite for ash.

EPA is currently modeling a range of treatment and disposal costs associated with K178 management. This range reflects uncertainty associated with whether or not ferric chloride residues exhibit a hazardous characteristic or not. If the residues are characteristic, then there is a pre-existing requirement to treat this material, including treatment for any dioxins and furans present. In this case, the only attributable cost from this rule would be the difference between Subtitle D nonhazardous landfill and Subtitle C hazardous waste landfill costs. This represents the lower bound of the cost range. If the ferric chloride residues are not characteristically hazardous, then all hazardous waste treatment and disposal costs are attributable to this rulemaking. This represents the upper bound of the cost range and include incineration costs of ferric chloride residues.

<u>Comment 22-10b:</u> Correction of incorrect assumptions

<u>The EPA assumption that only 10% of the chloride solids will be Bevill nonexempt (page 4-11) is not consistent with the overreaching proposed listing of wastes.</u>

65 FR 55762 states: "We propose to list as hazardous the non-exempt portion of the solid wastes generated from the production of titanium dioxide via the chloride-ilmenite process. This listing covers the non-exempt portions of the wastewater treatment solids generated at all three facilities, any non-exempt portions of the chlorinator solids (e.g., any mass derived from the vanadium wastes), and ferric chloride solids generated at the Delaware facility. To the extent that these listed materials remain commingled with solids that would otherwise be exempt, the entire commingled mass is subject to the listing (see § 261.3(b)(2)). "

Since the vast majority of the solids produced from the chloride ilmenite process are commingled with "masses derived from the vanadium wastes" and DuPont knows of no demonstrated technical way to separate the vanadium stream from these other streams, we must assume that all chloride-ilmenite solids will have to be treated and disposed of as K178 wastes.

The calculation from the scenario in the previous analysis must now be multiplied by a factor of 10 (100% versus the previous 10%) to provide the total cost.

10 x $88,065,780/yr = $880,657,800/yr annually

The "Economic Analysis" (pages 2-11, 4-15 & B-2) states that DuPont has Company sales of $26,918 million and Profits of $7,690 resulting in a profit margin of 28.6% (page B-5). The sales and net income were taken from the 1999 DuPont Annual Report and used as a basis for showing the insignificance of the calculated incremental costs to DuPont's income.

The Center for Economics Research overlooked the fact that $7,471 million of the Net Income was from the sale of an asset (Conoco). The proper number to use would be the "Income from Continuing Operations" of $219 million.

DuPont's calculated incremental impact of $88 million or $880 million is obviously a significant percentage of the Company profits.

The "Economic Analysis" (pages 4-9 & 4-14) calculates the estimated revenue for titanium dioxide sales to be $474 million per site. Based on a calculated incremental impact of $0.966 million EPA calculates the impact to DuPont's titanium dioxide business to be 0.20% of sales.

Based on DuPont's calculated incremental annual impact of $88 million for the 3 sites the additional cost to DuPont's titanium dioxide business is 6.2% of sales, which is a very significant percentage of profit.

Based on DuPont's calculated incremental impact of $880 million the additional cost to DuPont's titanium dioxide business is equivalent to 62% of sales.

Response 22-10b: EPA has clarified in today's final rule that it does not consider the vanadium oxychloride purification stream to be within the scope of the K178 listing.

Regarding measures for economic impact used in preparing the economic impacts estimates for the final rule, the Agency used Hoovers (www.hovers.com) to obtain financial information about DuPont. The Agency incorporated the reported sales and profits shown in this source into the final economic analysis.

Comment 22-10c: Using DuPont's actual production numbers, corrected for excluded costs and incorrect numbers:

The Agency's assumptions concerning the ore blend used overstates the percentage of solids that are actually produced. Using actual solids volumes from 1998 we calculate below what we believe to be the realistic incremental costs for treatment and disposal of the proposed listed wastes.

The "Economic Analysis" uses a 60% ilmenite ore (page 4-10) as the feedstock to the process. It is asserted (page 4-11, footnote 2) that the process will generate 4% chloride solids of which 10% will be Bevill nonexempt and thus require treatment.

Based on actual 1998 numbers for production of solids and approximated % ash values the following costs for loading, transportation, incineration, stabilization and disposal are calculated:

11,750 mt/yr x[($239.38 - $79.00) + 0.925 x ($238.36 - $53.20 + $93.11) + $727.51 + ($103.00 - $61.80)/20 + 0.925 x ($103.00/20 + $239.38)] = $16,139,095/yr (cumulative annual total)

Response 22-10c: Today's final rule lists as hazardous waste only ferric chloride residues associated with ferric chloride production. The estimated volume of this material generated was provided to EPA by DuPont in its letter of May 8, 2001. The estimate used in preparing the economic impact analysis for the proposal was based on published information available about Ti02 concentrations in ilmenite ore. We believe that the assumptions made in preparing the economic impact assessment in support of the proposal were appropriate, based upon available information.

Comment 22-10d: Using DuPont's actual production numbers, correction of incorrect assumptions

The EPA assumption that only 10% of the chloride solids will be Bevill nonexempt (page 4-11) is not consistent with the overreaching proposed fisting of wastes.

5 FR 55762 states: "We propose to list as hazardous the non-exempt portion of the solid wastes generated from the production of titanium dioxide via the chloride-ilmenite process. This listing covers the non-exempt portions of the wastewater treatment solids generated at all three facilities, any non-exempt portions of the chlorinator solids (e.g., any mass derived from the vanadium wastes), and ferric chloride solids generated at the Delaware facility. To the extent that these listed materials remain commingled with solids that would otherwise be exempt, the entire commingled mass is subject to the listing (see § 261.3(b)(2)). "

Since the vast majority of the solids produced from the chloride ilmenite process are commingled with "masses derived from the vanadium wastes" and DuPont knows of no demonstrated technical way to separate the vanadium stream from these other streams, we must assume that all chloride-ilmenite solids will have to be treated and disposed of as K178 wastes.

Based on actual numbers for production of solids and approximated % ash values the following costs for loading, transportation, incineration, stabilization and disposal are calculated:

164,200 mt/yr x[($239.38 - $79.00) + 0.812 x ($238.36 - $53.20 + $93.11) + $727.51 + ($103.00 - $61.80)/20 + 0.812 x ($103.00/20 + $239.38)] = $215,657,314/yr (cumulative annual total)

Response 22-10d: As stated above under Response 22-10b, EPA has clarified in today's final rule that it does not consider the vanadium oxychloride purification stream to be within the scope of the K178 listing. The ferric chloride solids are generated separately at the Edge Moor plant. It is reasonable to assume that DuPont can segregate them and manage them separately.

Comment 22-10e: The cost of retrofitting surface impoundments and construction of hazardous waste storage facilities was not included in the Agency's estimated costs.

DuPont estimates a minimum of $55 million to retrofit surface impoundments and construct hazardous waste storage facilities for the three facilities affected by the proposed rule. This type of cost was not considered in the "Economic Analysis" document and was thus not included in the Agency's estimated incremental costs. This additional number increases the cost, which needs to be included in the OMB analysis.

Response 22-10e: EPA believes that retrofitting surface impoundments to meet minimum technology standards does not represents the least-cost compliance alternative for DuPont. Rather, a least-cost regulatory response would be to install wastewater treatment tanks in lieu of retrofitting existing surface impoundments. EPA notes that it included the cost of installing wastewater treatment tanks in our cost estimate for the proposed economic analysis (see page 4-11 of August 2000 Economic Analysis for Listing of Inorganic Chemicals, Notice of Proposed Rulemaking). For surface impoundments to store hazardous waste, the units must be permitted and meet minimum technology requirements including double liners, leachate collection systems and leak detection, the costs associated with these requirements would be much higher than the cost associated with installing a tank-based system. DuPont stated in its letter to EPA of May 8, 2001 that the company is working with the State of Delaware regarding the closure of its remaining active surface impoundments.

Comment 22-11: DuPont Disagrees with the Agency's Questioning of the Legitimacy of the Delaware Facility's Ferric Chloride as a Product and the Reference of the Delaware Facility's Ferric Chloride Product as a Waste Acid.

Comment 22-11a: Ferric Chloride at the Delaware facility is a commercial product produced according to established product specifications.

On 65 FR 55759 EPA called the Delaware Facility's ferric chloride product stream a waste acid and DuPont disagrees with this nomenclature. Ferric Chloride is a commercial product produced at the Delaware facility and is not a waste. Ferric chloride is a co-product with Ti02 and is commercially marketed to the water and wastewater treatment industry. DuPont has and continues to invest capital dollars and dedicate technical resources to sustain ferric chloride as a competitively viable and environmentally sound product. DuPont has put considerable effort in making its product consistent or better than competitor's ferric chloride. Ferric chloride produced at the Delaware facility has been sold commercially since 1974, prior to enactment of the Resource Conservation and Recovery Act of 1976. This material has product specifications and is sold into the wastewater, potable water markets, and industrial wastewater treatment applications. As a result of the investment and commitment to this commercial business, DuPont has strengthened its sales position in the marketplace over the last 26 years.

DuPont strongly disagrees with EPA's statement questioning the legitimacy of the ferric chloride produced at the Delaware facility. The ferric chloride produced at Delaware facility has been sold commercially since 1974. In October 1994, DuPont entered a business partnership with a leading North American coagulant manufacturer and marketer providing them exclusive marketing rights to the ferric chloride produced at the Delaware facility.

The ferric chloride produced at the Delaware facility follows the established product specifications in accordance with the American Water Works Association (AWWA) Standards for liquid ferric chloride. Daily analytical testing is done to ensure compliance of these standards. The ferric chloride produced at the Delaware facility is sold at market value into several market segments, and the selection of ingredient ore for Ti02 manufacture includes consideration of the quality impact of ferric chloride co-product. In addition, DuPont continues to invest and reinvest in this commercial business with dedicated quality assurance resources, Research & Development resources and programs, and a Business Manager dedicated 100% to ensure that the ferric chloride produced at the Delaware facility meets the current and future market specifications.

Response 22-11a: See comment responses 12-5b, 22-3a-2, 8-4a, and 22-3a-2. EPA is not listing the ferric chloride sold as a product. Thus, EPA need not reach a conclusion about the legitimacy issue that the commenter raised. EPA notes that it is listing solids that separate during ferric chloride storage onsite.

Comment 22-11b: Ferric Chloride is a commonly used coagulant in the wastewater and water treatment industry.

As indicated previously, the material produced at the Delaware facility is sold to an inorganic coagulant market leader who also manufactures and sells a variety of inorganic coagulants including ferric chloride produced in Canada. The material produced at the Delaware facility is marketed in the same manner as their other products and supported by the same sales organization and infrastructure utilizing typical marketing and advertising vehicles including pamphlets and other sales aids, as well as participation at annual trade shows, exhibits and technical conferences (i.e., WEFTEC, AWWA, and Intertech). Sales brochures routinely reflect ferric chloride as a "time-tested product for higher quality water at low costs." It is recognized in the industry as a proven approach to meet tough standards and realize cost savings in the operations of water treatment plants while minimizing capital investments. Ferric chloride is used throughout the United States and Canada for overcoming problems in wastewater treatment plants. Market pricing, estimated shipments, production of ferric chloride, sales growth, and competitive analyses are reported in *Chemical Marketing Reporter, Current Industrial Reports,* and Chemical Economics Handbook, SRI International. *Ullmann's Encyclopedia of Industrial Chemistry,* and other miscellaneous industry and trade reports.

Ferric chloride produced at the Delaware facility has received National Sanitation Foundation (NSF) approval and is certified by NSF for use as drinking water treatment additive. The ferric chloride produced at the Delaware facility satisfies all the AWWA guidelines. The effective dose of ferric chloride is between 5 and 25 PPM, therefore any impurities present are well below regulatory levels even as a component of sludge associated with wastewater treatment. In addition, studies and customer results have shown that ferric chloride effectively coprecipitates heavy metals such as arsenic and selenium.

As published in April 1999 Chemical Economics Handbook by SRI International, ferric chloride whether it is produced at the Delaware facility or produced by a competitor, is primarily sold into the wastewater market segment for phosphate removal, chemically enhanced primary treatment, biosolids conditioning, heavy metal co-precipitation, sludge dewatering, struvite control, odor and corrosion control, and to coagulate suspended matter and neutralize surface charges. Ferric chloride's popularity as a water treatment chemical stems from its physical and economic properties. Ferric chloride's rapid hydrolysis in water aids in making it an ideal flocculating and precipitating agent. The ferric hydroxide produced forms flocs (small clumps) that adsorb suspended particles of various materials. The clumps, with the adsorbed matter, are then allowed to settle out for removal. Even though ferric chloride is generally priced comparatively to alum, on a per-pound of iron basis, ferric chloride delivers better performance. Dosage levels of the ferric chloride produced at the Delaware facility does not vary from ferric chloride produced by competitors, and is more efficient than aluminum based competitive products.

The Chemical Economics Handbook reports ferric chloride consumption for potable water treatment has increased significantly in the 1990's. The main reason for the growth is the acceptance of a new treatment process called enhanced coagulation which involves removing as much organic matter as possible before the chlorination step in a water treatment plant. Ferric chloride has been found more effective than alum in this end use, primarily because it forms more dense, less bulky sludge that is easier to remove. The Partnership for Safe Water is a voluntary cooperative effort between the EPA, AWWA and other drinking water organizations and as of April 1999, consists of 225 surface water utilities representing 330 water treatment plants throughout the United States. The goal of this cooperation is to provide a new measure of safety to millions of Americans by implementing prevention programs where legislation or regulation does not exist and are based on optimizing treatment plant performance and increasing protection against microbial contamination in America's drinking water supply. The Partnership for Safe Water has set a lower voluntary standard for turbidity of 0.1 NTU as opposed to 1 NTU. Using ferric chloride produced at the Delaware facility has enabled a major U.S. City to easily meet this standard and be recognized by the Partnership for Safe Water.

DuPont ferric chloride is also sold as coagulant in industrial wastewater treatment for the coprecipitation of heavy metals from wastewater effluent. It is also used to remove color such as humic matter from wastewater, for sludge dewatering, and to control hydrogen sulfide in industrial sludge conditioning applications.

Ferric chloride's use in coagulation and flocculation of fine particles to enhance settling and clarification in water and wastewater, and its use as a chemical conditioner of wastewater sludges, are so commonly practiced that it is discussed in virtually every text on the subject. More recent texts (i.e., those written within the past 25 years) also describe the use of ferric chloride to precipitate phosphates in wastewater in order to control nutrient-laden discharges that could cause eutrophication. The following are only a few referenced articles that document the use of ferric chloride in the treatment of water, wastewater, and wastewater sludge. Each is from a text considered a classic in the industry and/or by an author renowned in the field:

1) Chemical interaction in particle separation, by Werner Stumm, a peer-reviewed article from Environmental Science and Technology. Stumm was the co-author, along with James J. Morgan, of the most widely recognized water chemistry book, Aquatic Chemistry. The article describes principles of chemical coagulation and phosphate precipitation refers to ferric salt addition throughout the text.

2) Water Supply and Pollution Control, by Viessman and Hammer, the fourth editionof the classic general text by Clark, Viessman and Hammer. The use of ferric chloride is described in excerpts from Chapter 11 on common chemicals used in water and wastewater treatment, Chapter 12 on wastewater treatment odor control, and Chapter 14 on phosphate removal.

3) Wastewater Engineering: Treatment, Disposal, and Reuse (a.k.a. "Metcalf and Eddy"), the third edition of the wastewater "bible", revised by renowned environmental engineering professor George Tchobanoglous and Frank Burton, retired VP of Metcalf and Eddy. Chapters 7 and 11 on chemical precipitation - using ferric chloride either as a settling aid or to precipitate phosphate.

4) Physicochemical Processes for Water Quality Control, the classic text on process fundamentals edited by world-renowned professor Walter Weber. Specifically referencing the chapter on Coagulation and Flocculation written by Charles O'Melia, recognized as the leading expert in the subject in his time. Substantial discussion of the use of ferric chloride or iron (III) salts in general is provided.

5) Developing Industrial Water Pollution Control Programs: A Primer, by Wesley Eckenfelder, the most recognized name in the wastewater field. The chapter on Coagulation discusses the use of ferric chloride as a coagulant and for phosphorus removal.

6) Water: the universal solvent, the Nalco handbook. Nalco Chemical Company has been one of the leading producers of water and wastewater treatment chemicals. Included in the handbook is a table of common chemicals used to treat water, which features ferric chloride.

Clearly, ferric chloride is a commonly used product in the water and wastewater industry that is recognized by the world's experts for its effectiveness. Any assertions or implications that it is not a legitimate product are obviously misguided.

DuPont disagrees with the EPA's comments on page 55759 regarding the legitimacy of use of the ferric chloride produced at the Delaware facility for drinking water and wastewater treatment reagent based on metal content as well as trace amounts of chlorinated dioxins and furans. As mentioned previously, the metal content meets the AWWA and NSF standards established for drinking water and is not significantly different in total than competitive products used as water treatment reagent. Dioxin and furans are an unintended artifact of the chloride ilmenite and chloride TiO_2 process. Extremely low levels of dioxin can be found in the ferric chloride. DuPont's risk assessments and analysis indicate that the ferric chloride does not contribute in any measurable way to dioxin that might be detected in treated potable water.

Response 22-11b: See comment 22-11a.

Comment 22-11c: DuPont plans to close its impoundments. However, DuPont disagrees with EPA's view that impoundments should be associated with the discard of waste rather than the storage of product.

DuPont acknowledges the fact that at the Delaware facility it uses surface impoundments to store a portion of the ferric chloride prior to its sale as a water and wastewater treatment reagent. DuPont, however, disagrees with EPA's view that impoundments should be associated with the discard of waste rather than the storage of products. Although DuPont feels that the potential for ground-water release and exposure from the impoundments at the Delaware facility is relatively low as stated in EPA's 1990 Report to Congress on page 13-9, DuPont plans to close its impoundments. Over the past 8 years, DuPont has made significant progress in implementing new technology to move towards total tank-based processing/storage of its iron chloride product at its Delaware facility. In 1998, DuPont ceased using three out of four impoundments used to process/store iron chloride at its Delaware facility. A conceptual closure plan for these three impoundments was submitted to the regulating state agency in 1998. The remaining iron chloride impoundment is used as additional capacity to the Delaware facility's two tank based units. DuPont has met with the regulating state agency throughout this year in order to gain state agency approval by year's end of its closure plan for all four impoundments.

Response 22-11c: See comment 12-5b and 22-11a.

Comment 22-11d: The EPA should not make any reference to the legitimacy of the product in the drinking water and wastewater applications in their final ruling.

The EPA did not sample competitive ferric chloride, other coagulants, or treated potable water and therefore has no basis to differentiate the ferric chloride produced by the Delaware facility from competitive ferric chloride or non iron-salt coagulants on the basis of metal content and/or dioxin and furans. Significant volumes of ferric chloride have been produced and successfully marketed and consumed by drinking water and wastewater plants since 1974. In February 1982, Ohio EPA provided DuPont with written approval of the use of ferric chloride produced at the Delaware facility as a product for conditioning and treatment of potable water. Further, in 1987, EPA recognized DuPont's efforts in support of the commercialization of ferric chloride produced at the Delaware facility for use in drinking water and wastewater applications. DuPont does not believe that the EPA has a justifiable basis for questioning the legitimacy of such a long-standing product that meets product specifications and satisfies existing regulatory standards. The EPA should not make any reference to the legitimacy of the product in the drinking water and wastewater applications in their final ruling.

Response 22-11d: See comment 22-11a.