

Remediation of P₄ Contaminated Matrices at FMC, Pocatello, Idaho

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INTRODUCTION:

The purpose of this paper is to provide an independent assessment of technologies to remediate the P₄ contamination at the FMC Pocatello, Idaho site. P₄ remains at the site in capped wastes ponds and in soils from spills and process leaks at the former P₄ production, storage, and handling areas. Most notably, FMC estimates that between 580 to 5,500 tons of P₄ impacting 44,000 to 137,000 tons of soil (Figure 4-2 SRI Report June 2008) reside under the former Furnace Building and Slag Pits (RU 1 and RU 2) to a depth of at least 80 feet; the P₄ is in contact with the groundwater. P₄ residues are also present in underground pipes.

For this report, the open literature, including patents, was reviewed and inquiries sent to points-of-contact for various P₄ contaminated sites.

My experience with P₄ stems from a U.S. Army impact area where residues from P₄ ordnance were killing waterfowl (Racine et al. 1992). As part of this Army project, I developed and used methods for the sampling and analysis of P₄ contaminated soils, water, and tissue. I also developed methods for the monitoring of the remediation of the impact area. Documentation on this project is summarized in two annotated bibliographies (Nam et al. 1999, Walsh 2003). The P₄ contaminated sediments on the impact area are sporadically distributed at the points of detonation of WP ordnance to depths of up to 1 m. The P₄ contaminated matrices at FMC are quite different in that the mass of P₄ and depths of contamination are far greater than in the impact area. Also, risk to human health is greater at FMC due to contaminants from the manufacturing process in addition to the presence of P₄.

BACKGROUND:

Nomenclature and Chemistry:

The chemistry of phosphorus is extensive because phosphorus forms multiple bonds with carbon, nitrogen and oxygen. As a result, the nomenclature describing phosphorus compounds is equally extensive and many times ambiguous. Frequently, the term “phosphate” and “phosphorus” are used synonymously. Phosphates are compounds containing phosphorus in oxidation state +5 bonded to oxygen atoms (PO₄) and are also referred to as orthophosphate. This unfortunate blurring of terms leads to great confusion because phosphates have very different properties than elemental phosphorus. The least confusing approach is to use a chemical formula rather than a name.

Elemental phosphorus exists in several allotropes (i.e. white, red, black, violet). All allotropes melt to form the same liquid that consists of tetrahedral molecules of P_4 , known as white phosphorus. White phosphorus is colorless when pure, but frequently contains traces of red phosphorus that result in a slight yellow color, hence the term “yellow phosphorus” that is used interchangeably with “white phosphorus.” In this report, the term white phosphorus will be used to refer to elemental phosphorus with the molecular formula P_4 , the form of concern at FMC.

Reaction of P_4 with oxygen will yield phosphorus pentoxide (P_4O_{10}) (Greenwood and Earnshaw 1984). However, the reaction is not as straightforward as many believe. The vigorous spontaneous combustion of white phosphorus is so extraordinary that the fact that P_4 vapor can persist in the presence of oxygen is often ignored. The Nobel Prize for chemistry was awarded to Nikolai Nikolaevic Semenov for explaining why “phosphorous vapours may stay ... in contact with oxygen without even a trace of reaction processes being observed.” Semenov proved that the oxidation proceeds in the vapor phase via a branched chain reaction through various suboxides within certain limits of oxygen and phosphorus pressures. Given the above, the possible presence of P_4 vapor must be considered for conceptual site models and for risk assessments.

One of the suboxides is phosphorus trioxide P_4O_6 that forms in oxygen limiting conditions. Phosphorus trioxide is a solid with a melting point of 23.8°C and a relatively high vapor pressure. The obnoxious garlic like odor that emanates from white phosphorus contaminated matrices is attributed to phosphorus trioxide.

Also of interest is phosphine (PH_3), which is the product of the alkaline hydrolysis of P_4 and of the hydrolysis of phosphorus trioxide.

P_4 , P_4O_6 and PH_3 are all highly poisonous. In contrast, P_4O_{10} , which hydrolyzes in water to form phosphoric acid, is an irritant.

Releases of White Phosphorus into the Environment

Environmental contamination with white phosphorus has occurred at facilities that either produce or use white phosphorus, during railway accidents involving white phosphorus tank cars, and in areas where white phosphorus munitions have been filled or detonated. Thorough and publicly available documentation of the extent of these releases and their

associated ecological impacts are limited to just a few cases. These include two sites where the release of water containing suspended and dissolved white phosphorus from P₄ manufacturing facilities caused massive mortality of fish (Long Harbour, Placentia Bay, Newfoundland and Muscle Shoals, Alabama) (Idler 1981, Rosenblatt et al. 1973), and where residues from the detonation of white phosphorus ordnance caused significant mortality of animals (waterfowl at Eagle River Flats, AK and livestock in Great Britain (Stewart and Thomas 1930, Adams et al. 1942)).

Anytime that white phosphorus is released into the environment, injuries are likely to follow. Notable news stories within the last few years concerning white phosphorus are: the wartime use of WP munitions in Fellujah, Lebanon, and Gaza (The Times Online 2009); the injury of a construction worker in Orlando, FL when he uncovered a World War II era WP ordnance (Orlando Sentinel 2008); injuries from white phosphorus smoke following a train derailment in Ukraine (Associated Press 2007); the collapse of a phosphorus furnace in Yunnan, China that released 50 tons of white phosphorus (China Chemical Reporter 2007); the poisoning of waterfowl at an Albright and Wilson waste pond in Oldbury, England (Express and Star 2008); and controversy over plans to build a hospital on top of a capped landfill containing 150,000 tons of phosphorus-contaminated wastes from an Albright and Wilson plant in Portishead, England (This Is Bristol 2008). The purpose of mentioning the above occurrences is to emphasize that problems with P₄ are worldwide and that releases long ago are still causing problems.

White phosphorus manufacturing has (almost?) ceased in the United States. As of 2007, only one site (Monsanto/Solutia/P₄ Production) in Soda Springs, Idaho was still in operation (Jasinski 2007). Most white phosphorus production has moved to China. Former manufacturing plants in North America in addition to the FMC plant are listed in Table 1. FMC was the largest producer of white phosphorus in the United States. Toxic Release Inventory data from 1988 to 2003 shows that over 97% of the white phosphorus reported as “Total On-Site Releases to Land” were from Idaho, and almost all were from FMC (Appendix A). The list of manufacturing plants in Table 1 should be considered incomplete. Smaller manufacturing facilities existed many years ago when white phosphorus was used for the production of matches. A 1907 USGS publication states that the first “phosphorus works” in the US was by Moro Phillips in Philadelphia in the 1860s followed by JJ Allen’s

and Sons also in Philadelphia in 1891. These were followed by a General Chemical Company plant in Long Island, NY, and the American Phosphorus Company's plants in Mount Holly Springs and in Yorkhaven, PA. The legacy of the wastes from these plants would be an interesting topic for a research project that may be pertinent to FMC.

TREATMENT TECHNOLOGIES

Treatment technologies for white phosphorus wastes were reviewed in previous documents for or by the EPA. A report by Booz Allen Hamilton in 2002 focused on the crude phosphorus clarifier tank at the Rhodia Silver Bow Plant in Montana. The EPA Report EPA 542-R-03-013 published in 2003 focused on the ponds at FMC in Pocatello. Both reports point out that many bench-scale treatability studies have been conducted on white phosphorus wastes, but the technologies have not been demonstrated at the pilot or full scale level. This statement is still true. Appendix B is a list of patents that address wastes from the white phosphorus industry.

The report for the Silver Bow, Montana site evaluated technologies for: a) *containment*, b) *removal and phosphorus recovery*, and c) *removal and treatment*. The *containment* technologies were: soil cap, enhanced cap (flexible membrane liner), vertical (sheet pile or impermeable slurry wall) and horizontal barriers (grout injected into the soil beneath the P₄ wastes). The *removal and phosphorus recovery* technologies were: chemical (solvent) extraction, conversion to phosphoric acid in a small phosphoric acid plant to be built on-site, and distillation at the Monsanto/Solutia P₄ production plant in Soda Springs, Idaho. The *removal and treatment* technologies were: acid (nitric) oxidation, Zimpro® anoxic reaction, Zimpro® wet air oxidation, on-site incineration and off-site incineration. The report concluded that both the soil and enhanced caps, both Zimpro® reactions, and both on-site and off-site incineration were viable options. They also considered removal and distillation at the Monsanto/Solutia P₄ production plant a viable option.

For the 2003 EPA report on the waste ponds in Pocatello, information was gathered from almost all the facilities listed in Table 1. The technologies evaluated were solidification/stabilization, caustic hydrolysis, chemical oxidation, mechanical aeration, incineration, and thermal desorption. These same technologies were said to be identified in the Eastern Marchaud Flats Feasibility Study in 1996.

The most notable update to the information obtained for the 2003 EPA report is the failure of the attempted solidification/stabilization at Stauffer Chemical Plant in Tarpon Springs, FL (Appendix C). The solidification/stabilization was intended to prevent leaching of metals from the former waste ponds. However, a pilot scale test conducted in February 2006 resulted in a fire when the alkaline cement mixture intended for solidification came in contact with subsurface white phosphorus. In addition to the fire, phosphine gas was released. The new remedy for this site is a groundwater “cut off” wall that will be installed around the perimeter of the former waste ponds. Capping will also be used.

A soil-bentonite slurry wall and HDPE cap was designed and installed by BCI Engineers and Scientists at the Electro-Phos site near Mulberry, Florida (BCI 2008).

At the Rhodia Silver Bow Plant near Butte, Montana, at least some excavation of P₄-contaminated soils was conducted. The following quote is from USEPA Region 8 Docket No. RCRA-08-2004-0003: “Elemental phosphorus and phosphoric acid (collectively “Phosphorous Material”) observed during excavation, will be drummed at the excavated site, or Pipe with such phosphorus material (and other materials requiring cleaning) will be transferred to the Plant for cleaning, followed by drumming of the removed phosphorus material. Each drum containing elemental phosphorus will be sent offsite for incineration.” This excavation was to be completed by 31 October 2004. A letter dated 20 September 2005 from EPA Region 8 indicated that this work was completed. The former EPA contact for this project (Rosemary Rowe) directed me to the current EPA contact (Sara Sparks). Documents describing this excavation are in the Helena, Montana library. Rhodia proposes to cap the rest of the P₄ contaminated areas; however, the state of Montana wants the wastes removed.

Comments on Technologies

Capping

In situ-capping has been the most frequently used technology in the white phosphorus industry. The obvious disadvantage is the perpetual liability for tons of untreated ignitable, reactive and very toxic material. Caps require long term maintenance and monitoring. Adequate enforcement of land use restrictions far into the future cannot be guaranteed. Pressures for development where land is in high demand (e.g., England and in Florida) has

already resulted in potential relaxation of restrictions on building on capped P₄-contaminated sites.

Capping is being considered for an operable unit at Aberdeen Proving Ground, MD (EPA ID: MD2210020036). The site is called the “New O-Field” and was used for the demilitarization of chemical warfare material including white phosphorus in trenches. The Record of Decision was targeted for September 2008 and was reported to include a permeable cover over the trenches, a groundwater treatment barrier, a wetland treatment buffer, and a “bio-beneficial” sediment cover (Stachiw 2008).

Excavation and recovery or treatment of P₄

Excavation should be evaluated given the mass of P₄ that is estimated to reside below RU1 and RU2. If the estimates of the mass of P₄ in the subsurface are correct, the P₄ mass could persist indefinitely. Current costs and safety hazards associated with excavation under controlled conditions need to be evaluated in light of the beneficial elimination future hazards from uncontrolled exposure of P₄. Additionally, the commercial value of the recovered P₄ should be considered. Safe removal of P₄ can be accomplished by preventing the sublimation of solid P₄. The formation of P₄ vapors are limited by cold temperatures or a covering of water.

Depending on the volume of excavated soil and the mass of P₄ in the soil, there are many options to consider for either treatment or recovery. Appendix B lists some of the patented methods that have been described, but most have not been implemented.

Zimpro® Reactions

An obvious technology to consider at FMC are the Zimpro® reactions. As described in Booz Allen Hamilton (2002), there are two reaction schemes. The first is the anoxic reaction where wastes are mixed with a lime slurry at elevated temperature and pressure, the off gases containing phosphine are treated in a thermal combustor, and the reacted slurry is treated with stabilization methods to reduce metal mobility. The second is a wet oxidation technology that occurs at lower reaction temperature and pH and higher pressure. The reaction product is P₄O₁₀, so a thermal combustion unit is not used. The anoxic technology was chosen for a pilot scale plant that was evaluated by Zimpro. A full scale plant was built to treat the wastes generated by FMC and to treat the sludge in Pond 18 (Ed Greutert, personal communication). However, the plant was not operated because FMC shut down the

Pocatello manufacturing facility and paid a cash settlement to the Shoshone-Bannock Tribe to allow the waste ponds to be capped (Lacey 2004).

Separation of Meltable Solid

White phosphorus melts at 44°C. This relatively low melting point can be used to separate P₄ from high melting point solids such as excavated soil. Sturdivant et al. (2002 (Appendix B)) patented a method for “Removal of Elemental Phosphorus from Mixtures with other Solids.” This technology would be appropriate only for matrices that have significant mass of P₄ and that can be fed into the recovery unit. The solids that remain after the meltable P₄ has been recovered would likely require further treatment.

Low Temperature Thermal Desorption

Thermal desorption was evaluated in the 2003 EPA report. An additional factor that should be added to a future analysis is that the temperature range required to transfer the bulk of the P₄ to the vapor phase does not need to be as high as the temperature range that is typically used for hydrocarbons. Significant sublimation can occur between 30 and 44°C. Low temperature thermal desorption should be evaluated.

Landfarming

Excavated soil with residual (not readily ignitable) P₄ can be treated by simply placing the soil under conditions that allow the P₄ to sublime. This technology is analogous to landfarming techniques that are used for hydrocarbon-contaminated soils. Once in the vapor phase it will ultimately oxidize. A case study where this technology was used is the following:

In 1986, in Miamisburg, Ohio a tanker car containing 40,000 L of liquid (45°C) P₄ derailed and burst into flames next to a stream feeding the Great Miami River which leads to the Ohio River (Scoville et al. 1989). Most of the contaminated stream sediment was removed and treated by exposing the sediment on open-air asphalt pads. Each pad was approximately 2000 m² (0.5 acre) with curbs, and the contaminated sediment was placed on each pad to a depth of 15 to 20 cm. A tractor with a cultivator disc attachment turned the sediment to constantly expose the P₄. To increase the rate of decontamination, the sediment was heated by propane heater-blowers attached to the rear of the tractor. A 3 to 8% hydrogen peroxide solution was

added also, although it was probably not necessary. The sediment was treated for 12 to 24 hr, the amount of time required to reduce the P₄ to less than 10 mg/kg. At concentrations less than 10 mg/kg the material was not deemed to be ignitable. Approximately 5,700 m³ were treated and disposed (manner of disposal was not described). Air monitoring was conducted throughout the treatment process because houses were within 400 m (0.25 mile) of the treatment area.

In the above case study, “smoke” was generated. Open air smoke generation is probably not desirable at the FMC site. This technology should be considered for soils with P₄ concentrations that are sufficiently low that smoke generation is not significant.

In-Situ Thermal Remediation

In-situ decontamination of P₄-contaminated soils via sublimation of solid P₄ residue has been occurring at the FMC site. However, for in-situ decontamination to occur, the P₄ must have surface area exposed and a path for the P₄ vapor to diffuse to the atmosphere where it will eventually form phosphate. Oxidation products of P₄ that form in the soil pore space can encapsulate residual solid P₄ and it will remain in encapsulated until the oxidation products are removed. This situation is more likely to occur where the soil is dry and the mass of P₄ is greater than a few grams. We have found that moist sediments with P₄ do not form localized oxidation products. The sediment moisture provides a path for the diffusion of oxidation products. P₄ can also be encapsulated by structures such as piping, roads, or building foundations.

In-Situ Thermal Remediation is a technology used to remove organics and mercury source zones. Baker et al. (2006) reviews four methods: In-Situ Thermal Desorption, Steam-Enhanced Extraction, Electrical Resistance Heating, and Dynamic Underground Stripping. A description of this technology can be found at www.terratherm.com. The vapor pressure of P₄ is sufficiently high that In-Situ Thermal Desorption should be evaluated for the recovery of the large mass of P₄ under RU1 and RU2.

Piping Decontamination

The US military decommissions WP ordnance at Crane Army Ammunition Plant. The technology is the “APE 1400 White Phosphorus-Phosphoric Acid Conversion Plant (WP-PAC)”. The facility has operated since 1989 and has the capacity to consume 11,550

pounds of white phosphorus and produces 48,000 pounds of phosphoric acid (75%) a day (BMPCOE 2007, CAAA 2008). The resulting phosphoric acid and scrap metal are sold. This method may be applicable to the contaminated piping at Pocatello.

CONCLUSIONS

The FMC Pocatello plant was the largest P_4 manufacturing site in North America. Technologies to recover P_4 that were not feasible for smaller plants may be commercially viable at the Pocatello site due to the larger volumes of product that were lost during production (leaking sumps) or disposal in the waste stream. There are still significant unknowns at the FMC site including the actual volume of contaminated soils, the horizontal and vertical gradients in the concentrations of P_4 at RU1 and RU2, the total mass of potentially recoverable P_4 , and the form of P_4 in the soil (discrete nodules, veins in the subsurface, dispersed throughout the soil pore spaces?). The capped waste ponds, especially Pond 16s, present an enormous technological problem due to the uncontrolled chemical reactions that are producing phosphine. In the foreseeable future, the capped ponds will need considerable investment to prevent releases of hazardous gases and the formation of explosive conditions.

The most important decision will be whether to recover the bulk of the white phosphorus or to leave it in place. Excavation of soil and piping under cold climatic conditions (i.e., well below the auto-ignition temperature of P_4) should be carefully considered as an option. The ponds are different because of their unstable conditions, and are examples of the unforeseen consequences of leaving a reactive waste in place. Recovery of the wastes from under the caps should not be attempted until conditions stabilize. A combination of technologies will be required to address the complex situation in Pocatello.

Table 1: Other P₄ Manufacturing Sites in North America (Gleason (2007)).

Facility Name	City	State	Year Closed
U.S. TVA National Fertilizer Development Center	Muscle Shoals	Alabama	1976
Agrifos Nichols Plant	Nichols	Florida	
Exxon Mobil ElectroPhos Division	Mulberry	Florida	1983
Stauffer Chemical Company (Victor Chemical)	Tarpon Springs	Florida	1981
Monsanto Chemical Company (Solutia/P4 Production)	Soda Springs	Idaho	still open in 2007
Stauffer Chemical (Rhone-Poulenc/Rhodia, Inc.)	Silver Bow	Montana	1997
Oldbury Electrochemical Company (Albright and Wilson/Hooker Electrochemicals Inc.)	Niagara Falls	New York	
Monsanto Chemical	Columbia	Tennessee	1986
Shea Chemical/Hooker Chemical/Occidental Chemical Company	Columbia	Tennessee	
Stauffer Chemical Company (Victor Chemical Works/Rhone-Poulenc)	Mt. Pleasant	Tennessee	1991
Albright and Wilson America Limited (Tenneco)s	Varenes	Quebec	1993
Albright and Wilson (Electric Reduction Company of Canada Industries Limited (ERCO))	Buckingham	Quebec	1957
Albright and Wilson America Limited (Rhodia) Phosphorus Plant (Electric Reduction Company of Canada Industries Limited (ERCO))	Long Harbor	Newfoundland	1989

Note: The chemical companies that operated the P₄ manufacturing sites in North America have been in flux. The following is an example: "Rhodia, a Delaware corporation with its principal place of business in Cranbury, New Jersey, acquired the Silver Bow Site from Rhone-Poulenc Inc. ("RPI") pursuant to an Asset Contribution Agreement ("ACA"), dated January 1, 1998. The legal successor to RPI is Defendant Bayer, a New York corporation with its principal place of business in Research Triangle Park, North Carolina. Compl.. At the time of Rhodia's acquisition of the Silver Bow site, both Rhodia and RPI were controlled by Rhone-Poulenc S.A. ("RPSA"). Aventis, a French corporation with its principal place of business in Strasbourg, France, became the legal successor to RPSA in December 1999. In December 2004, Aventis merged into and with Sanofi, a French corporation with its principal place of business in Paris, France" From United States District Court District OF New Jersey Case 2:04-cv-06424-GEB-MF Document 46 Filed 11/07/2007

Table 2. Toxic Release Inventory Reported Total On-Site Releases (pounds) of White Phosphorus to Land for Idaho and the United States 1988-2006 (www.epa.gov/tri).

Year	Idaho	United States	Idaho/US (%)
1988	3,864,100	3,925,604	98%
1989	3,278,360	3,309,430	99%
1990	2,189,313	2,225,076	98%
1991	1,914,389	1,944,585	98%
1992	1,881,386	1,918,765	98%
1993	1,720,076	1,765,757	97%
1994	1,699,491	1,746,241	97%
1995	1,871,795	1,904,083	98%
1996	2,042,574	2,069,480	99%
1997	2,581,457	2,606,675	99%
1998	2,272,970	2,300,483	99%
1999	2,715,468	2,805,586	97%
2000	979,338	985,841	99%
2001	542,545	616,073	88%
2002	160,575	165,698	97%
2003	212,315	218,133	97%
2004	860	18,897	4.6%
2005	172	119,359	0.1%
2006	446	50,489	0.9%

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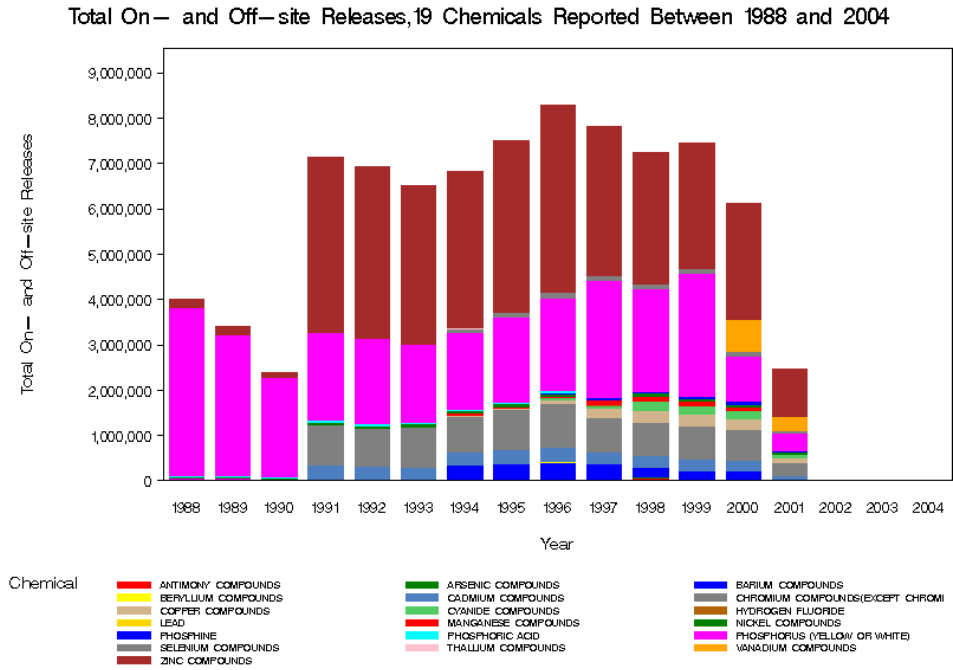
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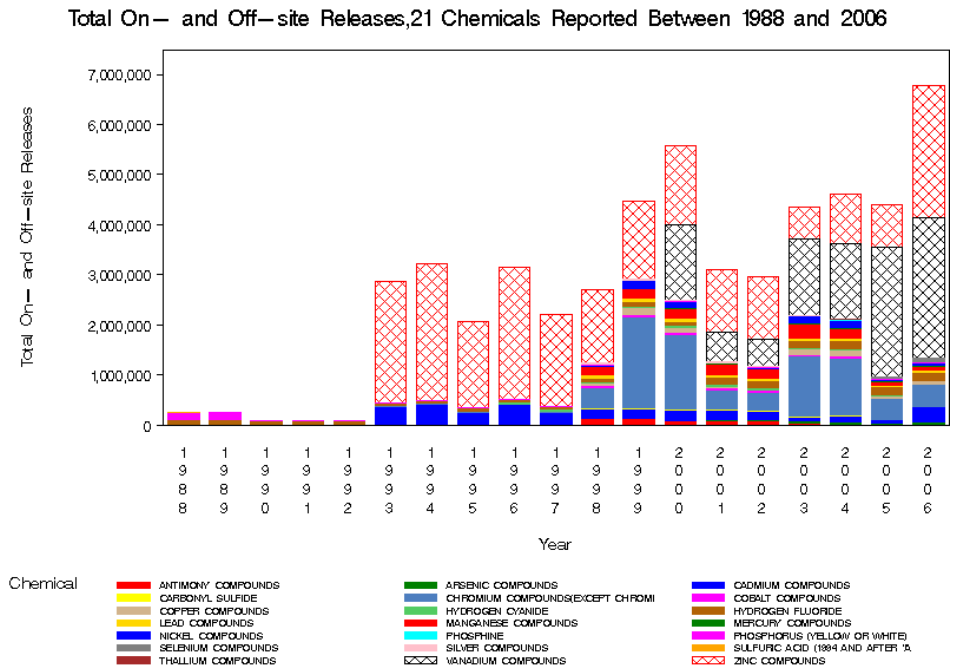
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Appendix A. Toxic Release Inventory Data Trend Graphs from www.epa.gov/tri

a) FMC Idaho LLC (TRI Facility ID Number 83202FMCCR3MIWE)



b) P4 Production LLC (TRI Facility ID Number 83276MNSNTHIGHW)



Appendix B. Patents for treating wastes from the white phosphorus industry.

Patent No.	Date	Title	Inventor
US 6955796 B2	18 Oct 2005	Process for Total Environmental Remediation of a Phosphorus Contaminated Site	Mohan S. Saran and George W. Luxbacher
US 2004/0241073 A1	2 Dec 2004	A Process for Total Environmental Remediation of a Phosphorus Contaminated Site	Mohan S. Saran and George W. Luxbacher
US 6620396 B2	16 Sep 2003	Method of Removing Phosphorus from Sludge	Mohan S. Saran
US 6451276 B1	17 Sep 2002	Recovery of Elemental Phosphorus from Phosphorus Sludge	Mohan S. Saran and others
US 6446812 B1	10 Sep 2002	Removal of Elemental Phosphorus from Mixtures with other Solids	Charles N. Sturdivant and others
US 6169222B1	2 Jan 2001	Remediation of Soil Polluted with Phosphorus-Containing Wastes	James C. Barber
6051201	18 Apr 2000	Preparation of Phosphatic Feedstock from Phosphorus-Containing Waste	James C. Barber
5549878	27 Aug 1996	Processes for the Disposal and Recovery of Phosphy Water	James C. Barber
5275639	4 Jan 1994	Recovery of Phosphates from Elemental Phosphorus Bearing Wastes	Jack M. Sullivan and others
4961912	9 Oct 1990	Passivation of Elemental Phosphorus Contained in Waste Ponds	Auston K. Roberts and others
4761271	2 Aug 1988	Method for Treating Destabilized, Fire-Involved Elemental Phosphorus	Arthur D.F. Toy and Fred Jaffee
4686094	11 Aug 1987	Treatment of Pyrophoric Elemental Phosphorus-Containing Material	Auston K. Roberts and others
4492627	8 Jan 1985	Recovery of Phosphorus from Waste Ponds	David A. Crea
4402833	6 Sep 1983	Waste Water Treatment System for Elemental Phosphorous Removal	W. Clark Bennett and Theodore T. Garrett
4284515	18 Aug 1981	Process for Decreasing Elemental Phosphorus Levels in an Aqueous Medium	Chong T. Liu
4206925	27 May 1980	Recovery of Phosphorus from Sludge	Miguel Coll-Palagos
US 6921520 B2	26 Jul 2005	Process for Upgrading Raw Phosphate Ore	James C. Barber

Appendix C: Attachment (Stauffer_TarponSprings_June2007.pdf). Explanation of Significant Differences Fact Sheet. Stauffer Chemical Superfund Site, Tarpon Springs, Pinellas County, Florida, June 2007