United States
Environmental Protection
Agency

Office of
Solid Waste
Washington, D.C. 20460

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Background Document for Identification and Listing of the Deferred Dye and Pigment Wastes

Note: Due to business confidentiality concerns, we have removed information from this document, if the information may potentially disclose information claimed as confidential business information (CBI). We note the missing information in the text, where appropriate. See section 1.2 for further discussion.

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1.0 INTRODUCTION

This background document summarizes the findings and data used to support proposed regulations for wastes generated by certain processes in the dye and pigment manufacturing industry.

The U.S. Environmental Protection Agency's (EPA's) Office of Solid Waste (OSW), as directed by Congress in the Hazardous and Solid Waste Amendments of 1984 to the Resource Conservation and Recovery Act (RCRA), has undertaken an investigation of the wastes generated by the Dyes and Pigments Industry. This investigation also was mandated by a 1991 consent decree resulting from litigation brought against the Agency by the Environmental Defense Fund (EDF).

As a result of the consent decree, the Agency conducted an industry study to identify and characterize wastes from dyes and pigments manufacturing processes. There are three major classes of dyes and pigments: azo/benzidine, anthraquinone, and triarylmethane. The settlement agreement specifies that the Agency investigate the following dye and pigment categories for possible hazardous waste listing:

- azo, monoazo, diazo, triazo, polyazo, azoic, and benzidine categories of the azo/benzidine dye and pigment class
- anthraquinone and perylene categories of the anthraquinone dye and pigment classes
- triarylmethane, triphenylmethane, and pyrazolone categories of the triarylmethane dye and pigment class

In addition, the settlement agreement specifies that the following types of wastes be investigated:

- spent catalysts
- reactor still overheads
- vacuum system condensate
- process waters
- spent adsorbent
- equipment cleaning sludge
- product mother liquor
- product standardization filter cake
- dust collector filter fines
- recovery still bottoms
- treated wastewater effluent
- wastewater treatment sludge

1.1 Dyes and Pigments Proposed Listing Determination in 1994

On December 22, 1994 (59 *FR* 66071) the Agency proposed listing determinations for five wastes; a no-list determination for six wastes; and deferred action on three wastes. The five wastes proposed to be added to the list of hazardous waste are:

1. K162 Wastewater treatment sludge from the production of azo pigments.
2. K163 Wastewaters from the production of azo pigments.
3. K164 Wastewater treatment sludge from the production of azo dyes, excluding FD&C colorants.
4. K165 Wastewaters from the production of azo dyes, excluding FD&C colorants.
5. K166 Still bottoms or heavy ends from the production of triarylmethane dyes or pigments.

A final listing determination on these wastes is still pending.

Also on December 22, 1994, the Agency proposed that six additional wastes not be listed. The six wastes that had a no-list determination are:

- Wastewaters from the production of triarylmethane dyes and pigments (excluding triarylmethane pigments using aniline as a feedstock).
- Wastewater treatment sludge from the production of triarylmethane pigments using aniline as a feedstock.
- Wastewaters from the production of triarylmethane pigments using aniline as a feedstock.
- Wastewaters from the production of anthraquinone dyes and pigments.
- Wastewaters from the production of FD&C colorants.
- Dusts and dust collector fines from the manufacture of dyes and pigments.

The Agency also deferred action on three waste streams based on insufficient characterization data, or lack of health-based levels for specific constituents of concern. The "deferred" dye and pigment waste streams are the subject of this background document. The three deferred wastes are:

- Spent filter aids, diatomaceous earth, or adsorbents used in the production of azo, anthraquinone, or triarylmethane dyes, pigments, or FD&C colorants.
- Wastewater treatment sludge from the production of triarylmethane dyes and pigments (excluding triarylmethane pigments using aniline as a feedstock).
- Wastewater treatment sludge from the production of anthraquinone dyes and pigments.

This document will refer to these wastes as "filter aids," "TAM sludges," and "anthraquinone sludges" respectively.

The Agency deferred a determination as to whether to list Filter Aids in 1994 due to insufficient waste characterization data for this widely variable waste (see 59 *FR* 66103; December 22, 1994).

As described in the 1994 proposed rule, EPA evaluated wastes from the production of TAM pigments that use aniline as starting material ("feedstock") separately from other TAM wastewaters and wastewater treatment sludges. This was because the process that uses aniline as a feedstock is somewhat different (see 59 *FR* 66081 and 66096).[1] We proposed listing decisions for wastes from TAM dyes and pigments derived from aniline in the 1994 notice, but deferred a decision for wastewater treatment sludge from the production of TAM dyes and pigments that do *not* use aniline. This document addresses the wastewater treatment sludges from production of TAM dyes and pigments, excluding TAM pigments using aniline as a feedstock.

EPA deferred a listing determination for anthraquinone dye and pigment sludges in 1994. In our analysis of EPA collected samples, we could not detect any constituents attributable to the production of anthraquinone dyes or pigments. Data supplied by industry indicate the presence of two constituents on EPA's analyte list, however no health-based benchmarks exist for these chemicals. In the 1994 rule, we requested any information that commenters may have on the toxicity of these constituents (see 59 *FR* 66101).

1.2 Confidentiality Claims

For the purpose of developing the supporting data for listing rulemakings for the dye and pigment industry, a questionnaire was sent out to industry pursuant to RCRA Section 3007. Some of the information collected from industry and used in the 1994 proposed rule, as well as in the current effort, was claimed as confidential. As a result of a consent order and a subsequent preliminary injunction in connection with a case brought by some of the dye and pigment industry to prevent the disclosure of information claimed as confidential business information (CBI), Magruder et al. v. U.S. EPA, Civ. No. 94-5768 (D.N.J.), the EPA is enjoined from disclosing information claimed as confidential until all CBI determinations have been made on the data intended to be published in connection with these proposed rules.

Therefore, as with the 1994 proposed rule, we have removed information from this document, if the information may potentially disclose information claimed as CBI. We note the missing information in the text, where appropriate. However, we have included data that are not claimed as CBI, whenever such data are available. We have also included data that we obtained from public or non-CBI sources.

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[1]See Lewis, A.P. (Ed), *Pigment Handbook*, Vol. I, *Properties and Economics*, Wiley-Interscience, New York, 1988, pp. 587-599.

2.0 Industry Description

The dye and pigment industries are comprised of three separate type of manufacturers, dye manufacturers, pigment manufacturers, and Food, Drug, and Cosmetic (FD&C) colorant manufacturers. The trade association representing the dye industry, the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry (ETAD), defines dyes as "intensely colored or fluorescent organic substances which impart color to a substrate by selective absorption of light." When a dye is applied, it penetrates the substrate in a soluble form, after which it may or may not become insoluble. Dyes are retained in the substrate by physical absorption, salt or metal-complex formation, solution, mechanical retention, or by the formation of ionic or covalent chemical bonds.[2]

Dyes are used to color fabrics, leather, paper, ink, lacquers, varnishes, plastics, cosmetics, and some food items. According to literature published by ETAD, dye manufacture in the U.S. includes more than 2,000 individual dyes, the majority of which are produced in quantities of less than 50,000 pounds.[3] The U.S. International Trade Commission's (USITC) production data for the five-year period from 1990 through 1994 indicate that dye production was highest in 1993 at approximately 160,000 tons. Production declined in 1994 to approximately 156,000 tons. More recent production information is not available.

Pigments possess unique characteristics that distinguish them from dyes and other colorants. The Color Pigment Manufacturers' Association (CPMA), the trade association representing the manufacturers of pigments in the U.S. and Canada, defines pigments as "colored, black, white, or fluorescent particulate organic or inorganic solids which usually are insoluble in, and essentially physically and chemically unaffected by, the vehicle or substrate in which they are incorporated." CPMA explains that the primary difference between pigments and dyes is that, during the application process, pigments are insoluble in the substrate. Pigments also retain a crystalline or particulate structure and impart color by selective absorption or by scattering of light. With dyes, the structure is temporarily altered during the application process, and imparts color only by selective absorption.

Pigments are used in a variety of applications; the primary use is in printing inks. There are fewer pigments produced than dyes, though pigment batches are generally larger in size. The USITC indicates that total U.S. production of organic pigments was an estimated 71,500 tons in 1995.[4]

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[2] "Dyes and Dye Intermediates." *Kirk-Othmer Encyclopedia of Chemical Technology*, Fourth Edition. Volume 8. New York: John Wiley & Sons, Inc, 1993.

[3] *Pollution Prevention in the Dye Manufacturing Industry*, Ecological and Toxicological Association of Dyes and Organic Pigments Manufacturers, U.S. Dye Manufacturers Operating Committee of ETAD, October, 1994.

[4] *Industry and Trade Summary: Synthetic Organic Pigments*. U.S. International Trade Commission. Publication 3021. February, 1997.

FD&C colorants are dyes and pigments that have been approved by the Food and Drug Administration (FDA) for use in food items, drugs, and/or cosmetics. Typically, FD&C colorants are azo or triarylmethane dyes and are similar or identical to larger-volume dye products not used in food, drugs, and cosmetics. Manufacture of FD&C colorants is similar to that for the corresponding dye or pigment, except that the colorant undergoes additional purification. Each FD&C colorant batch is tested and certified by the FDA.[5]

The end-user markets for dyes and pigments, which include textiles, paper, leather, ink, paints, coatings, plastics, fibers, and other low volume markets, are not included in this listing evaluation. Consistent with both HSWA Amendments of 1984 and the consent decree, EPA is only making proposed determinations on wastes from the production and manufacturing of dyes and pigments. Therefore, wastes associated with dye or pigment use that are generated beyond the manufacturing facility fall outside of the proposed listing definitions.

There are different ways to classify organic dyes and pigments. One method is by chemical structure or class. The EPA's current study of the dye and pigment industries is directed at three chemical classes of organic dyes and pigments: azos, anthraquinones, and triarylmethanes. Azos are the largest and most versatile chemical class. The various azo chemical structures are readily synthesized, typical product application methods are generally not complex, and a broad range of colors can be produced with excellent fastness properties. Azos are used in essentially all organic dye applications, and azo colorants exist for dyeing all natural and synthetic substrates.[6]

Anthraquinones are often within the vat and disperse dye chemical classes. Despite high costs, anthraquinone vat dyes are an important group of dyes due to superior fastness. Their main application is on cotton and cellulose fibers. Disperse anthraquinone dyes are used in coloring hydrophobic, synthetic fabrics. They have good affinity for the substrate, level dyeing power, and excellent fastness.[7]

Triarylmethanes are characterized by their brilliancy of hue, intensity of color, and low fastness properties. Triarylmethanes are typically used in the production of pigments or printing and duplicating inks. In addition, this class of dyes is used the textile industry, primarily on synthetic fibers.[8]

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[5] "Food Dyes." *Colour Index*, Third Edition. The Society of Dyers and Colourists and the American Association of Textile Chemists and Colorists. 1971.

[6] "Azo Dyes." *Kirk-Othmer Encyclopedia of Chemical Technology*, Fourth Edition. Volume 3. New York: John Wiley & Sons, Inc, 1993.

[7] "Anthraquinone." *Kirk-Othmer Encyclopedia of Chemical Technology*, Fourth Edition. Volume 3. New York: John Wiley & Sons, Inc, 1993.

[8] "Triphenylmethane and Related Dyes." *Kirk-Othmer Encyclopedia of Chemical Technology*, Fourth Edition. Volume 24. New York: John Wiley & Sons, Inc, 1993.

Another common method of classifying dyes is based on the application process. Common application process classes include: acid, basic, direct, reactive, disperse, vat, and solvent. Acid dyes are water-soluble, anionic dyes used primarily on nylon, wool, silk, and modified acrylics. Basic, direct, and reactive dyes are also ionic, water-soluble compounds. Basic dyes are cationic dyes that are generally used to dye modified acrylics, nylons, and polyesters. Direct dyes, which are generally anionic azo compounds, are applied to cellulose fibers in the presence of electrolytes. Reactive dyes, developed for application to cellulosic fibers such as cotton, contain a reactive component that forms a covalent bond with the fiber, resulting in high color fastness.[9]

Disperse, vat, and solvent dyes, as well as pigments, are insoluble in water. Disperse dyes are applied to hydrophobic fibers such as cellulose acetate, nylon, polyesters, and acrylics from a fine aqueous dispersion. Solvent dyes are soluble in an organic solvent and generally are used for varnishes, lacquers, inks, and various synthetic materials.

The dye and pigment industries typically operate successive batch processes producing varying dye and pigment products. These batch operations generate a wide variety of solid wastes on a periodic basis. These wastes generally can be divided into two general types: commingled wastes and process-specific wastes (see Exhibit 3-1). Commingled wastes are wastes combined from multiple processes prior to management (e.g., wastewaters). Commingled wastes include secondary wastes generated from the treatment of other commingled wastes (e.g., wastewater treatment sludges). Process-specific wastes are wastes that are unique to a specific process and may be managed independently of other wastes (e.g., spent filter aids).[10] [11]

2.1 RCRA Section 3007 Survey Questionnaire

EPA's Office of Solid Waste developed a detailed RCRA §3007 questionnaire for distribution to dye and pigment manufacturing facilities (see the 1994 proposed rule docket No. F-94-DPLP-FFFFF, item S0266). EPA sought feedback from industry trade and made appropriate modifications to the survey instrument prior to its distribution. The Agency distributed the questionnaire to approximately 110 domestic manufacturers in March of 1992. In order to determine the universe of facilities in the dye and pigment manufacturing industry a variety of reference books, trade associations, and individuals were utilized. Specifically, the following sources were used: 1988 U.S. International Trade Commission Report "Synthetic Organic Chemicals, U.S. Production and Sales"; 1987 Directory of Chemical Producers; and the *Colour Index*. In addition, ETAD and the Dry Color Manufacturers' Association (now the Color Pigment

[9]"Dyes and Dye Intermediates." *Kirk-Othmer Encyclopedia of Chemical Technology*, Fourth Edition. Volume 8. New York: John Wiley & Sons, Inc, 1993.

[10]Ibid

[11]*Pollution Prevention Guidance Manual for the Dye Manufacturing Industry*. U.S. Environmental Protection Agency and the Ecological and Toxicological Association of the Dyestuffs Manufacturing Industry.

Manufacturers' Association, CPMA) were contacted. The final information sources were industry representatives who were contacted by telephone.

The purpose of the questionnaire was to collect information on the 12 specific residuals identified in the 1991 consent decree. Most questions in this survey requested information on waste generation and management activities in 1991.

From data provided by questionnaire respondents, a number of facilities [the precise number was removed due to CBI concerns] were found to manufacture azo, anthraquinone, or triarylmethane dyes or pigments. The remaining facilities were either found not to manufacture dyes or pigments within the three classes under evaluation, or they were found not to manufacture any dyes or pigments. The Agency reviewed the questionnaire responses and requested follow-up information from many facilities. The scope of the questionnaire is discussed below.

2.1.1 Scope of the 1992 RCRA §3007 Questionnaire

In the questionnaire EPA collected information regarding the type of plant operations (manufacture of dyes and/or pigments, manufacture of dye and/or pigment intermediates, compounding and/or repackaging of dye and/or pigment products, and manufacture of other products). This information was used to determine the scope of facilities affected by the industry study.

EPA also collected information regarding the products manufactured at the facility. Information included product name, chemical structure of the product, raw materials and additives used in its manufacture, 1991 production volumes, and other information.

While the questionnaire requested information regarding the chemical characterization of wastes generated (e.g., any known or expected contaminants and their concentrations), such data were limited. The questionnaire did not require respondents to develop or collect information not readily available to characterize the wastes. Thus, EPA relied on its sampling and analysis to collect information for waste evaluation.

The questionnaire also collected information on the management of the wastes generated by each facility, including waste quantity and how the wastes were managed and/or disposed. EPA used this information to define the scope of the residual waste codes and the impact of listing on these facilities.

2.1.2 Updating Questionnaire Data for the Deferred Wastes

EPA contacted companies in this industry to update the information in the §3007 survey for the three deferred wastes at issue. The updated information EPA collected includes the quantities of wastes generated (for the year 1997), and the waste management practice used by the facilities for each of the deferred wastes.

2.2 Site Visits

In support of the 1994 proposed rule, the Agency performed site visits at facilities to observe facility waste generation and management practices and collected samples of wastes for chemical analysis. To characterize the wastes generated at dye and pigment manufacturing facilities, EPA collected samples from facilities to characterize the 12 residuals evaluated by the Agency. Appendix A summarizes the constituents found in all wastes. The number of samples and facilities are removed due to business confidentiality concerns. The dye and pigment manufacturers also provided a limited amount of additional waste sampling and analysis data in 1994. These additional data include aggregated analytical results from 19 industry analyses of samples that EPA and the facilities split during sampling visits. An industry trade group (Color Pigment Manufacturers' Association, or CPMA) aggregated this analytical information and submitted this information to EPA in April 1994. CPMA also included this information in the group's public comments on the 1994 proposed rule (see Docket No. F-94-DPLP-FFFFF, item DPLP-0025). We used the available sampling data from these sources to identify potential constituents of concern (see Section 4.0).

3.0 Waste Description, Quantities, and Management Practices

3.1 Spent Filter Aids, Diatomaceous Earth, and Adsorbents used in the Production of Azo, Anthraquinone, and Triarylmethane Dyes, Pigments and FD&C Colorants

Manufacturers add filter aids (e.g., diatomaceous earth) to some reaction processes to remove particulate impurities.[12] The spent filter aids then are collected in a filter press and the press cake, sometimes called a clarification sludge, is discharged as waste. In some cases, facilities also use filter aids following completed reactions to clarify and purify certain products (e.g., activated carbon). A typical process that generates filter aid materials is shown in Exhibit 3-1 (from the 3007 questionnaire EPA sent to industry). The Agency grouped spent filter aids, diatomaceous earth, and adsorbents used in the production of all relevant classes of dyes and pigments, because these wastes typically adsorb unreacted raw materials, by-products, and impurities. The constituent composition of these filter aids is expected to vary depending on dye/pigment produced and the raw materials used.

3.1.1 Waste Quantities and Management Practices

In response to the 1992 questionnaire, dye and pigment manufacturers reported generating filter aid wastes; the number of facilities and total waste quantity in 1991 are not included due to business confidentiality concerns. For the same reason, we have not included the updated data collected by EPA in 1998. Facilities that generated spent filter aids may generate this waste from the production of a wide variety of different dyes and pigments. For example, one facility (which did not claim the waste quantities as CBI) reported generating a total of 90 Mtons of filter aid wastes in 1997, comprised of 18 filter aids arising from the production of different dyes and/or pigments. The quantities and management practices for spent filter aids are summarized in Exhibit 3-2; further details are given in Appendix B.

[12] Diatomaceous earth has many commercial names (Filtercel, Celite, Supercel).

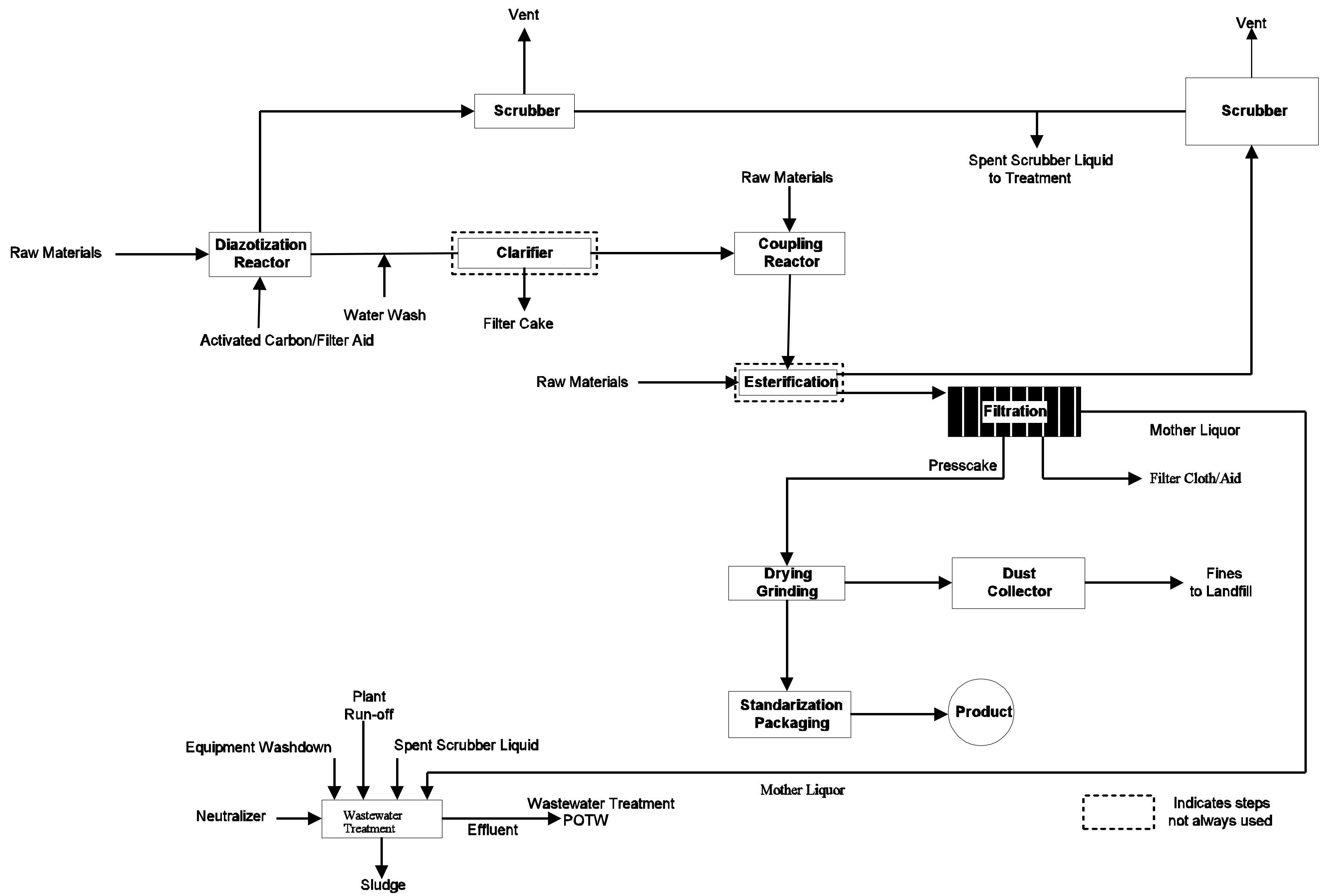

Exhibit 3-1. Typical Process for Dye or Pigment Production

Exhibit 3-2: Updated Generation Statistics for Filter Aids, Diatomaceous Earth, and Adsorbents Used in the Production of Azo, Anthraquinone, and Triarylmethane Dyes, Pigments, and FD&C Colorants, 1997[1]				
Final Management	**Number of Facilities[2]**	**Total Volume (Mtons)**	**Median Volume (Mtons)**	**90th Percentile Volume (Mtons)**
Offsite Municipal Landfill	**	**	**	**
Incineration	1	2.3		--
**				
**				
**				
TOTAL**				

[1]From waste data summarized in Appendix B.
[2]One facility reported two practices.
**Information removed due to business confidentiality concerns. Two facilities that reported waste volumes without CBI claims sent wastes to municipal landfills (108 Mtons and 90.4 Mtons).

3.1.2 Waste Characterization

The limited data on waste constituents for spent filter aids are shown in Appendix C (removed due to business confidentiality concerns). Because of the limited data available and the widely variation expected for these wastes, EPA developed a concentration-based listing approach for this waste. EPA chose constituents of concern based on the analytical data for *all* waste samples, combined with the reported use of chemicals as materials in the production of dye and pigments. This is described in detail in later in this document.

3.2 Wastewater Treatment Sludge from the Production of Triarylmethane Dyes and Pigments (Excluding Triarylmethane Pigments Using Aniline as a Feedstock)

A typical triarylmethane manufacturing process includes a condensation reaction, usually between an aromatic amine and an aromatic aldehyde. Once complete, the reaction products are then oxidized, sometimes with the aid of a catalyst. Additional reactions may be required following the addition of other raw materials.[13] Like most dye and pigment products, the product is typically sent to a clarification filter prior to finishing and packaging. Process wastewaters are treated to yield a wastewater treatment sludge.

[13]*Triarylmethane Colouring Matters,* page 4379, in the *Colour Index International,* CD-ROM, 3rd edition, the Society of Dyers and Colourists and the American Association of Textile Chemists and Colorists. 1996.

Wastewater treatment sludge is usually generated via the treatment of the following process waste streams: equipment washdown, plant run-off, spent scrubber liquid and mother liquor. The typical wastewater treatment sludge is generated from neutralization to adjust pH, clarification, and biological treatment. Sludge streams are further processed, typically through filtration and dewatering, prior to disposal. EPA deferred any listing decision for TAM sludges due to insufficient waste characterization data (see 59 *FR* 66095). Waste management practice details are removed due to business confidentiality concerns.

3.2.1 Waste Quantities and Management Practices

In response to the 1992 questionnaire, dye and pigment manufacturers reported generating TAM wastewater treatment sludges; the number of facilities and total waste quantity are removed due to business confidentiality concerns. For the same reason, we have not included the updated data collected by EPA in 1998. Waste quantities and management practices are summarized in Exhibit 3-2; further details are given in Appendix B.

3.2.2 Waste Characterization

In support of the 1994 rule, we attempted to sample TAM sludges (from production of TAM pigments that do not use aniline as a feedstock), however, the facility was not manufacturing TAM dyes or pigments during our sampling visit. Thus, we could not attribute any constituents detected in these samples to TAMs production.

Exhibit 3-2: Updated Generation Statistics for Wastewater Treatment Sludge from the Production of Triarylmethane Dyes and Pigments, 1997[1]

Final Management	Number of Facilities	Total Volume (Mtons)	Median Volume (Mtons)	90th Percentile Volume (Mtons)
Offsite Municipal Landfill	**	**	**	57[2]
**				-
TOTAL**				

[1]From waste data summarized in Appendix B.
[2]Due to limited data, EPA used this volume reported by one facility in modeling. One facility did not claim the waste quantity (57.2 Mtons) nor the practice of disposal in a municipal landfill as CBI.
**Information removed due to business confidentiality concerns.

3.3 Wastewater Treatment Sludge from the Production of Anthraquinone Dyes and Pigments

The anthraquinone (AQ) dye and pigment class includes anthraquinone and perylene. The production process involves reaction of an anthraquinone derivative to form the desired colored anthraquinone. The typical anthraquinone sludge is generated via the treatment of process wastewater similar to that described for TAM sludges.

3.3. Waste Quantities and Management Practices

We are not including information on the number of facilities generating this waste, nor the waste quantities reported for 1991, due to business confidentiality concerns. We also are not including information collected by EPA in 1998 on the number of generators and the quantities for 1997 for the same reason.

3.3.1 Waste Characterization

As noted in the 1994 proposal (see 56 *FR* 66101), EPA's analysis of anthraquinone sludges did not yield any contaminants that EPA could attribute to anthraquinone production. However, industry data submitted showed the presence of two constituents of potential concern. (The names of these constituents are not included in this document due to business confidentiality concerns on the underlying industry analytical data).

Exhibit 3-3: Updated generation Statistics for Wastewater Treatment Sludge from the Production of Anthraquinone Dyes and Pigments.

Final Management	Number of Facilities[1]	Total Volume (Mtons)	Median Volume (Mtons)	90th Percentile Volume (Mtons)
**				
**				--
TOTAL**				

[1]One facility reported two practices.
**Information removed due to business confidentiality concerns.

4.0 Constituents of Concern

As discussed earlier in this document and in the 1994 proposed rule, EPA found technical difficulties in obtaining representative waste characterization data for two of the deferred waste streams, filter aids and TAM sludges. EPA developed a concentration-based listing that does not rely on sampling and analysis of the two specific wastes at issue. In a concentration-based listing, EPA establishes concentration levels ("listing levels") for constituents of concern that define whether a waste from a specific generator would be hazardous. For this approach, EPA calculated listing levels for all constituents which can reasonably be expected to be found in the wastes. The Agency developed a list of constituents of potential concern using several data sources, and this section of the background document describes the methodology used.

4.1 Source of Constituents Considered for Inclusion as Potential Constituents of Concern

As noted above, the Agency obtained waste analysis data from its own analysis of samples of wastes generated at dye and pigment manufacturing facilities, and also from analytical results from industry analyses of samples that EPA and facilities split during EPA's sampling visits. We used the available sampling data from all these sources to identify potential constituents of concern.

The initial universe of constituents of potential concern included any constituent detected in any wastestream from the production of all classes of dyes and pigments EPA examined (i.e., all wastes sampled associated with the production of azo, TAM, and anthraquinone dyes or pigments). The broad universe of constituents detected in all waste sample provides an appropriate starting point for selecting constituents of concern for filter aids, because these wastes may be generated from the production many kinds of dyes and pigments. Because TAM sludge is a much more narrowly defined waste, EPA relied on materials used in TAM production to determine what constituents we expect in the waste.

4.2 Choosing Core Constituents of Concern

EPA used the analytical data to develop an initial list of potential chemicals of concern. We then reduced and augmented this list based on several factors. First, we can only develop a concentration level if a health benchmark exists for the chemical. Therefore, we removed constituents without health benchmarks from further evaluation. The sources we used for health benchmark data are summarized below; the Risk Assessment Background Document[14] in the docket for this rule contains further information. The remaining possible constituents of concern with health-based benchmarks are shown in Table 4-1.

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[14] *Development of Risk-Based Listing Concentrations for Hazardous Constituents Contained in Spent Filter Aids and Triarylmethane (TAM)Wastewater Treatment Sludges* Background Document, Appendix E, June, 1999.

Due to the lack of health-based benchmarks, we excluded certain constituents from consideration that we previously evaluated for azo dye and pigment wastes in the 1994 proposed rule. These constituents are acetoacetanilide (AAA), acetoacet-o-toluidine (AAOT), and acetoacet-o-anisidine (AAOA). For the 1994 proposal, we derived health based numbers for AAA, AAOT, and AAOA based on a Structural Activity Relationship (SAR) analysis. The Agency has since reevaluated and revised the SAR analysis based on comments received in response to the 1994 proposal. The revised analysis, which has been independently peer reviewed, concludes that the current available data are insufficient to make a quantitative estimation of the carcinogenic potential of these compounds, or to establish provisional non-cancer benchmarks. The revised toxicological analysis for these compounds and the peer review documents are provided in the Risk Assessment Background Document.[15]

While EPA analyzed samples from a few facilities for metals, the Agency dropped metals from any further evaluation as constituents of concern due the inability to link them directly to production of organic dyes and pigments. From the 3007 Survey, EPA found no reported use of metals in any significant quantity in the production process for the organic dyes and pigments under investigation, except for three metals. We are not including information on the identity of these chemicals due to business confidentiality concerns. EPA does not believe these chemicals would be useful as criteria for listing wastes from dye and pigment production; discussion of EPA's reasoning is not included due to business confidentiality concerns. [In any case, EPA completed preliminary risk analyses for selected metals to confirm that these metals are not likely to be of concern in these wastes. See the Risk Assessment Background Document for further details.]

EPA screened the potential constituents in Table 4-1 to remove chemicals that of little use in defining the hazardous characteristic of the two wastes at issue. In this analysis we considered the prevalence with which a constituent is used in the manufacturing of the different classes of dyes and pigments at issue in the consent decree, the likelihood that a chemical could be attributed to such production, and the frequency with which a chemical was detected in wastes samples. In considering if the constituents detected are likely to be derived from dye or pigment production, we used publicly available information from the Colour Index.[16] For example, we retained any chemicals that were detected that are commonly used as raw materials in the production of the dyes and pigments at issue (e.g., aniline is widely used in the production of azo products; see Colour Index, vol. 4, pages 4009 and 4699). We also kept some chemicals detected that have no apparent use as raw materials, because they may be impurities or degradation products from chemicals used in the manufacturing process (e.g., naphthalene may be an impurity in a commonly used raw material, beta-naphthol). We removed some constituents, such as acetone and

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[15]*Development of Risk-Based Listing Concentrations for Hazardous Constituents Contained in Spent Filter Aids and Triarylmethane (TAM)Wastewater Treatment Sludges* Background Document, Appendix A, June, 1999.

[16]*Colour Index International*, CD-ROM, 3rd edition, the Society of Dyers and Colourists and the American Association of Textile Chemists and Colorists. 1996.

methylene chloride, that were detected in samples, because they are common laboratory contaminants and/or common solvents that have no reported use in the production of these dyes and pigments. While such constituents may be present in wastes, we did not consider them further because we could not reasonably attribute them to dye and pigment production processes sampled. We dropped other constituents that had little or no reported use in the Colour Index; the dropped constituents were also rarely found in waste samples. EPA's reasoning in retaining or discarding a potential constituent is summarized in Table 4-1.

Table 4-1 summarizes EPA's evaluation of the chemicals for use in a concentration-based listing for filter aids and TAM sludges. EPA classified the chemicals in one of four categories: (1) a core constituent for spent filter aids, (2) a core constituent for TAM sludges, (3) other constituents that EPA considers unlikely, but presents in the proposed rule for comment as a possible constituent, and (4) a constituent that was dropped.

Table 4-1 summarizes the frequency with which we detected the chemicals in waste samples, and prevalence of use of the chemicals in the production of the three dye and pigment classes (azo, TAM, and anthraquinone) as found in the *Colour Index*. For filter aids we included constituents on the final list of constituents of concern if we detected the chemicals with at least a low frequency (i.e, in more than one sample), and we found some evidence that industry used the chemicals in the production of the dyes and pigments at issue. We also selected several chemicals that we believe may be degradation products of other raw materials (e.g., p-phenylenediamine), or possible impurities in other starting materials (e.g., naphthalene). Finally, we included several compounds that may arise from TAM production, as described below, even though we do not have analytical data showing these chemicals are present in wastes from this industry.

We chose to add two chemicals for consideration as constituents of concern that were reported to be used in the production of TAM products, even though we did not find them in any waste samples. In the case of benzaldehyde, we did not analyze any of the wastes for this compound. However, this chemical is reported to be used in the production of TAM products (see *Colour Index*, vol.4, page 4727). We analyzed for the other chemical (the identity is not given due to business confidentiality concerns), but we did not find it in any samples. However, the 3007 Survey indicated significant use of this chemical in the production of TAM dyes. EPA did not succeed in obtaining waste samples during the production of TAM dye and pigments (excluding TAM pigments using aniline as a feedstock). Therefore, based on the known uses in TAM manufacturing, we considered these two chemicals as potential constituents of concern.

The publicly available information EPA used was consistent with the information provided by industry in responses to the 3007 Survey, except in a few cases. In some instances (the identities are not given due to business confidentiality concerns), the *Colour Index* showed use of these chemicals that was not confirmed in the 3007 Survey. Conversely, in the case of another chemical (the identity is not given due to business confidentiality concerns), the 3007 Survey indicated significant use in the production of TAM dyes, while the *Colour Index* did not. In cases where we had these discrepancies, we relied on the source that showed use and assumed that these chemicals may be used in production.

In choosing core constituents of concern for a concentration-based listing for filter aids and TAM sludges, we considered adding constituents from the "other possible" category shown in Table 4-1. We considered these chemicals because they were detected with a moderate frequency, they had some use in manufacturing the dye and pigment products of concern, or they are in a class of compounds that have been historically linked to dye production (e.g., benzidines). However, these constituents are unlikely to be present in these two specific wastes at levels of concern. Some of the chemicals in this category could not be linked to the production of the dye and pigment classes at issue. We did not include chemicals in the final list of core constituents of concern unless we could find some evidence that the presence of a chemical was related to the production of the classes of the dyes and pigments of interest (for filter aids, the production of azo, TAM, or anthraquinone products; for TAM sludges, the production of TAM products, excluding TAM pigments using aniline as a feedstock). This is because many waste samples were wastewaters or sludges collected from combined wastewater treatment systems, and such systems typically receive waste streams from various other production processes at facilities. We did not include other chemicals because they were never or rarely detected in any waste samples.

Table 4-1 Possible Constituents of Concern in Dye and Pigment Wastes

Constituent	Category				Frequency of Detection		Use In Production of Dye and Pigment Classes of Concern[3] **	Comments
	Core Filter Aid	Core TAM	Other Possible	Drop	EPA Data[1]	Industry Data[2]		
Acetone				x	High	S	No use	Rare use in other dye production (indigoid);[3] common lab solvent and contaminant
Aniline	x				High	S	High use (Azo); some use in TAM pigment (aniline based)	
Benzaldehyde	x	x			NA	Not reported	Moderate use in TAM products	Not analyzed, but common reactant in TAM production[3]
Benzene			x		Moderate	S	No use	No reported use; common contaminant from varied industrial sources.
Benzidine			x		Low	Not reported	No reported use domestically	Used historically (Azo), but no current domestic use found;[3] only detected in wastes from production that does not use filter aids
Benzyl alcohol				x	Rare	S	No use	Rarely detected, no use.
Bis(2-ethylhexyl)phthalate				x	Low	S	No use	Common contaminant from plastic
Bromodichloromethane				x	**	S	No use	Possible contaminant from treated water source.
Bromoform				x	ND	S	No use	No use; not detected in EPA samples
Chloroaniline, p-	x				Moderate	S	Rare use (Azo)	Aromatic amine; some use in other dye products (aminoketones)[3]
Chlorobenzene			x		Moderate	S	No use	Other uses as solvent.

Constituent	**Category**				**Frequency of Detection**		**Use In Production of Dye and Pigment Classes of Concern**[3] **	**Comments**
	Core Filter Aid	Core TAM	Other Possible	Drop	EPA Data[1]	Industry Data[2]		
Chloroform			x		Moderate	S	No use	Possible contaminant from treated water supplies; other uses as solvent
Chloromethane				x	Low	Not reported	No use	No known use for dye classes of concern; rare use in other dye production (methines)[3]
Cresol, p-	x				Moderate	S	Low use (Azo) found in Colour Index	Industry analysis of split samples did not distinguish the meta and para isomers
Dichlorobenzene, 1,4-				x	Low	Not reported	No use	No use
Dichlorobenzene, 1,2-			x		Moderate	Other	No use	Solvent uses in other dye production (Oxazine dyes)[3]
Dimethoxybenzidine, 3,3'-	x				Low/Moderate	S	Moderate use (Azo)	
Dimethylaniline, N,N-	x	x			Rare	Not reported	Moderate to high use found for TAM dye production; rare use otherwise	Possibly not detected due to lack of data from TAM wastes
Dimethylaniline, 2,4-			x		**	S (listed as present but not reported in specific samples)	Low use (Azo)	**
Diphenylamine	x	x			Moderate	Not reported	Low use (Azo); and rare use in TAMs	Indistinguishable from N-Nitrosodiphenylamine by EPA method (GC/MS).
Diphenylhydrazine,1,2- (or Azobenzene)	x				Moderate	Not reported	No use	Possible oxidation product of aniline; indistinguishable from azobenzene by GC/MS analysis
Ethylbenzene				x	Low	S	No use	Other uses as solvent

Constituent	**Category**				**Frequency of Detection**		**Use In Production of Dye and Pigment Classes of Concern[3] ****	**Comments**
	Core Filter Aid	Core TAM	Other Possible	Drop	EPA Data[1]	Industry Data[2]		
Formaldehyde	x	x			Moderate	S	Moderate use for TAM; low use for others	
Methyl Isobutyl Ketone (4-Methyl-2-pentanone)				x	Low	S	No use	Other uses as solvent
Methyl Ethyl Ketone (2-Butanone)				x	ND	S	No use	Other uses as solvent
Methylene Chloride				x	Moderate	Not reported	No use	Common lab solvent and contaminant
Naphthalene	x				Mod/High		No use	Possible impurity in common Azo raw material (beta-naphthol)
Phenol	x				Mod/High	S	Moderate use (Azo)	
Phenylenediamine, o- (2-aminoaniline)			x		Unknown	Not reported	Low use (anthraquinone)	EPA found analytical methods unreliable (very poor recoveries); may be indistinguishable from p-isomer
Phenylenediamine, p- (4-aminoaniline)	x				Low/Mod	Not reported	Moderate use (Azo)	Possible hydrolysis product of azo raw materials (aminoacetoacetanilide); indistinguishable from o-isomer in EPA analysis
Pyrene				x	**	S	No use	No use
Pyridine				x	Rare	Not reported	No use	No known use for dye classes of concern; rare use in other dye production (oxazine);[3] rarely detected
Styrene				x	ND	S	No use	No use; not detected in EPA samples

Constituent	**Category**				**Frequency of Detection**		**Use In Production of Dye and Pigment Classes of Concern[3] ****	**Comments**
	Core Filter Aid	Core TAM	Other Possible	Drop	EPA Data[1]	Industry Data[2]		
Toluene				x	Low	S	No use	Other uses as solvent; low toxicity
Toluidine, 5-nitro-o- (2-Methyl-5-nitroaniline)			x		ND	S	Moderate use (Azo)	Only found in one split sample; not found in any azo wastes by EPA
Toluidine, o- (2-aminotoluene)	x	x			Moderate	S	Moderate use (Azo); low use (TAM)	Hydrolysis product of raw materials (AAOT); EPA could not separate o- and p-isomers during analysis
Toluidine, p- (4-aminotoluene)	x	x			Moderate	S	Low uses (Azo and TAM)	EPA could not separate o- and p-isomers during analysis
Trichlorobenzene, 1,2,4-				x	Moderate	S	No use	No use
Xylenes				x	Low	S	No use	No use; low toxicity
**								

[1]As found in analysis of samples of all dye and pigment wastes by EPA; NA=not analyzed, ND=not detected.
[2]S = reported in split sampling data submitted by CPMA (see Appendix A).
[3]Use in production of azo, TAM, or anthraquinone products from the *Colour Index International*, CD-ROM, 3rd edition, the Society of Dyers and Colourists and the American Association of Textile Chemists and Colorists. 1996.
**Relevant data are not included at the present time due to business confidentiality concerns; all information for 17 constituents has been removed.

Using analytical data from all dye and pigment wastes sampled is clearly appropriate for filter aids. This is because filter aids are used to treat and purify a wide variety of wastes derived from all of the classes of dye and pigment products (azo, triarylmethane, and anthraquinone). The shorter list for TAM sludges also is appropriate, due to the more limited set of potential waste constituents for this single dye and pigment class.

Analytical Issues

EPA found problems in its analysis of dye and pigment waste samples for some of the constituents in Table 4-1 In a few cases, our analyses could not distinguish between two compounds when we used the usual EPA methods for semivolatile organic chemicals, GC/MS method 8270B in *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods;* SW-846, hereafter called SW-846). We found problems for four pairs of compounds: diphenylamine/N-nitrosodiphenylamine; 1,2-diphenylhydrazine/azobenzene; o-toluidine/p-toluidine; and o-phenylenediamine/p-phenylenediamine.

N-Nitrosodiphenylamine breaks down to diphenylamine in the method we used (EPA Method 8270B from SW 846). Therefore, these two chemicals could not be distinguished. This means that a detection by this method could be due to either compound being in the waste. We found no reported use of the N-nitroso-derivative, but we did find diphenylamine has some use in the production of azo and TAM products. Therefore, for this pair we selected diphenylamine as the likely constituent of concern. This means that generators would analyze for this constituent and assume any concentration measured is diphenylamine.

Similarly, we could not distinguish the compounds 1,2-diphenylhydrazine and azobenzene in the analytical method used, because these chemicals interconvert readily under analytical conditions. Thus, our data showed that one or both of these compounds were present in waste samples, but we could not tell which one. In this case, we did not find any reported use of either chemical in dye or pigment production processes. However we believe that the presence of either may be explained by oxidation of aniline from processes that use aniline as a reactant. Thus, in this case we are including the constituent with the lower concentration level (1,2-diphenylhydrazine) to be protective. This means that generators would analyze their waste for the total level of 1,2-diphenylhydrazine and/or azobenzene, and assume that the amount detected is due only to the more toxic 1,2-diphenylhydrazine. This is also reasonable because these compounds may readily interconvert in wastes or the environment, thus it is prudent to set the listing level for the more toxic component Azobenzene is the oxidized form of the 1,2-diphenylhydrazine and exists in a redox equilibrium under varying environmental conditions. The reduced azo group on the 1,2-diphenylhydrazine is easily amenable to oxidation and results in the formation of azobenzene. The inter-convertibility of 1,2-

diphenylhydrazine to azobenzene is widely reported in the literature, and occurs in environmental matrices such as soil and water.[17]

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In our analysis we also could not separate two isomers of toluidine (o-toluidine and p-toluidine) and phenylenediamine (o-phenylenediamine and p-phenylenediamine). While it may be possible to distinguish these isomers using other analytical methods, we have no data at this time to indicate this. For the toluidine isomers, we are proposing to include both isomers as constituents of concern. If generators cannot separate these isomers, they could determine a total quantity for both. In the absence of information on which isomer is expected in the waste, generators would assume that the measured concentration is due to the more toxic o-toluidine. Generators could use their knowledge of their production processes, however, to definitively rule out the presence of one of the isomers, and in this way identify which isomer is present. For example, if generators know that only one isomer is used in any of the relevant processes, they could document this and claim this as part of their determination.

For the o- and p-phenylenediamine isomers, we reviewed the analytical data and now believe that o-phenylenediamine cannot be reliably measured. We found that we could not reliably recover the chemical from samples with known concentrations (spiked samples). In addition, the reported usage of o-phenylenediamine in the production of dyes and pigments is relatively limited compared to the use of p-phenylenediamine (see *Colour Index,* vol. 4, page 4822). Furthermore, p-phenylenediamine may also form from the degradation of a widely used azo precursor, p-aminoacetoacetanilide. Therefore, because of these analytical problems, and also because its use in dye or pigment production is limited, we are not including o-phenylenediamine in the list of constituents of concern for either waste. Thus, generators would be required only to determine if p-phenylenediamine is present in their wastes below the listing levels.

[17] See for example, Lyman W.J., et al (1990). Handbook of Chemical Property Estimation Methods, American Chemical Society, Washington DC: p. 4-9; Pellai P. et al. (1982), Chemosphere, 11:299-317; Meylan, W.M. and Howard P.H. (1991), Environ. Toxicol. Chem., 10: 1283-1293; and Callahan M.A., Slimak, M.W., Gabel, N.W., et al. (1979), *Water Related Fate of 129 Priority Pollutants*. Volume II (USEPA-440/4-79-029b).

Appendix A. Summary of Analytical Data for All Dye and Pigment Wastes
[Some Information was removed from this Appendix due to business confidentiality concerns]

Summary of Analytical Data for All Dye and Pigment Wastes

Chemical	EPA Sampling Data[1]			CPMA Split Samples[2]	Other Industry Data[3] **
	non-CBI	CBI **	CBI and non-CBI**		
Acetoacet-o-toluidide (AAOT)				1/19	
Acetoacet-o-anisidide (AAOA)				2/19	
Acetoacetanilide (AAA)				reported[4]	
Acetone	6/10			11/19	
Aminoanthraquinone[5]				2/19	
Aniline	9/10			11/19	
Benzene	3/10			4/19	
Benzidine	2/10				
Benzyl alcohol	1/10			1/19	
Bis(2-ethylhexyl)phthalate[6]	1/10			1/19	
Bromoform				1/19	
Carbon disulfide				2/19	
Chlorobenzene	4/10			6/19	
Chloroethane				1/19	
Chloroform	2/10			3/19	
Chloromethane	1/10				
Dibromomethane				2/19	
Diphenylamine/N-Nitrosodiphenylamine	3/10				
Ethylbenzene	1/10			3/19	
Formaldehyde	3/10			2/19	
Isopropylamine				1/19	
Methylene chloride	2/10			4/19	
Naphthalene	2/10				
Phenol	4/10			5/19	
Pyrene				2/19	
Pyridine	1/10				
Styrene				1/19	
Trichloroethene	1/10				
Toluene	2/10			6/19	
1,2,3-Trichlorobenzene	1/10				
1,2,4-Trichlorobenzene	3/10				
Xylenes (total)	1/10			5/19	
N,N-Dimethylaniline	1/10				

Chemical	EPA Sampling Data[1]			CPMA Split Samples[2]	Other Industry Data[3] **
	non-CBI	CBI **	CBI and non-CBI**		
1-Naphthylamine	1/10				
1,2-Dichlorobenzene	3/10				
1,4-Dichlorobenzene	1/10				
1,2-Diphenylhydrazine/ Azobenzene	3/10				
2 & 4-Aminoaniline/ 2-Methoxyaniline	1/10				
2-, 3-, & 4-Aminotoluene	1/10				
2/ 4-Aminotoluene	2/10			4/19	
2-Aminotolune[7]				3/19	
4-Aminotoluene[8]				1/19	
2-Bromo-4,6-dinitroaniline				1/19	
2-Butanone				5/19	
2-Chloro-4-nitroaniline	1/10				
2-Hexanone				1/19	
2-Hydroxynaphthalene				2/19	
2-Methoxybenzenamine				3/19	
4-Methylphenol	2/10				
3/ 4-Methylphenol				3/19	
4-Nitroaniline	1/10				
2-Nitrophenol	1/10				
2,4-Dimethylaniline				reported[4]	
2,4-Dinitroaniline				1/19	
3-Aminoacetanilide	3/10			1/19	
3,3-Dichlorobenzidine				2/19	
3,3-Dimethoxybenzidine	3/10				
4-Chloroaniline	4/10			2/19	
4-Methyl-2-pentanone (Methyl isobutyl ketone)	2/10			2/19	
5-Nitro-o-toluidine				1/19	

[1]Frequency the constituent was found in EPA analysis of samples of all dye and pigment wastes from production of azo, TAM, and anthraquinone dyes/pigments and FD& C colorants (from *Listing Background Document: Hazardous Waste Listing Determination for the Dye and Pigment Industries*; Appendix D, November 30, 1994). Only data from samples which were not claimed as CBI are presented due to business confidentiality concerns.
[2]From data reported in a letter to EPA from Color Pigment Manufacturers' Association (CPMA), dated April 20, 1994, which presented aggregated data from split samples. CPMA also included this information in public comments on the 1994 proposed rule (see Docket No. F-94-DPLP-FFFFF, item DPLP-0025).
[3]Other industry data for samples of one waste; the data has been removed due to business confidentiality concerns.
[4]Reported in list of substances identified, but frequency not shown in specific samples.

[5]Reported as 1-aminoanthraquinone and unspecified isomer (aminoanthraquinone) in CPMA data.
[6]Same chemical as Diethylhexylphthalate (DEHP) reported in CPMA data.
[7]Also known as 2-toluidine or o-toluidine.
[8]Also known as 4-toluidine or p-toluidine.
**Information removed due to business confidentiality concerns; 32 constituents derived from data with CBI claims were removed.

Appendix B. Summary of Waste Quantities and Management Practices
[Some information was removed from this Appendix due to business confidentiality concerns.]

Spent Filter Aid --Wastes Quantities and Management**

Facility Name/Location	Waste Quantity[1] (metric tons)		Waste Management		Notes
	1992 (for '91)	1998 (for '97)	1992	1998	
**	**	90.44	**	Subtitle D	
Dye Specialties Jersey City, NJ	250	107.95	Subtitle D	Subtitle D	
Eastman Kodak Rochester, NY	0.57	2.31	Incinerate	Incinerate	

[1]Waste quantities are total for all filter aids generated and managed by cited practice.
**Information removed for some facilities due to business confidentiality concerns.

Wastewater Treatment Sludge Waste from the Production of Triarylmethane (TAM) Dyes and Pigments (Excluding TAM Pigments Using Aniline as a Feedstock)--Wastes Quantities and Management**

Facility Name/Location	**Waste Volume** (metric tons)		**Waste Management**		**Notes**
	1992 (for '91)	1998 (for '97)	1992 (for '91)	1998 (for '97)	
Dye Specialties Jersey City, NJ	78.9*	57.15*	Subtitle D Landfill (see notes)	Subtitle D Landfill	In 1998 response, company indicated EPA incorrectly reported '92 waste management–disposal was in a D landfill (not C landfill).

* indicates dedicated wastewater treatment (i.e., not commingled)
**Information removed for some facilities due to business confidentiality concerns

Wastewater Treatment Sludge from the Production of Anthraquinone Dyes and Pigments--Waste Quantities and Practice

[Information removed due to business confidentiality concerns]

Appendix C. Spent Filter Aid Waste Analysis Results
[Information in this Appendix removed due to business confidentiality concerns]