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Preliminary Study of Carbon Disulfide Discharges from Cellulose Products Manufacturers

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1.0 STUDYING DISCHARGES FROM REGENERATED CELLULOSE MANUFACTURERS

1.1 Introduction

EPA is studying wastewater discharges from regenerated cellulose manufacturers because these facilities report large discharges of carbon disulfide (CS₂) (U.S. EPA, 2006, U.S. EPA, 2010). Currently no existing regulation sets federal effluent limitations for CS₂ discharges from the seven regenerated cellulose manufacturers that operate in the United States. Cellulose products manufacturers use CS₂ in the viscose process to break down aged alkali cellulose prior to regeneration. CS₂ can enter the wastewater stream at these facilities from CS₂ unloading and storage, the viscose process, CS₂ recovery, and/or air pollution control.

The main pollutant of concern discharged from these facilities, CS₂, is a volatile organic compound. EPA researched how the CS₂ enters the wastewater and its fate in the facilities' existing wastewater treatment systems. EPA also collected data on whether the CS₂ discharges would impact human health or aquatic life. EPA continues to resolve the overall question of how significant concentrations of CS₂ remain in the wastewater, although CS₂ is highly volatile.

1.2 Background – Manufacturing Process

Regenerated cellulose products manufacturers use the viscose process to form cellulose film, sponge, food casings, and rayon from dissolving-grade wood pulp. In the viscose process, sheets of dissolving-grade cellulose pulp are saturated with caustic (e.g., sodium hydroxide) to convert the cellulose into alkali cellulose. The alkali cellulose partially oxidizes and degrades by aging in ambient air. Gaseous CS₂ is mixed with the aged alkali cellulose in a vessel to form sodium cellulose xanthate. The sodium cellulose xanthate is dissolved in aqueous caustic solution, creating the viscose solution. The viscose solution is ripened, filtered, degassed, and extruded, and then sulphuric acid is added to the viscose solution to form regenerated cellulose (Schmidtke, 2000). Figure 1 presents a simplified flow diagram of the general viscose process and the different process steps for each type of regenerated cellulose (cellulose film or cellophane, sponge, food casings, and rayon) (Schmidtke, 2000).

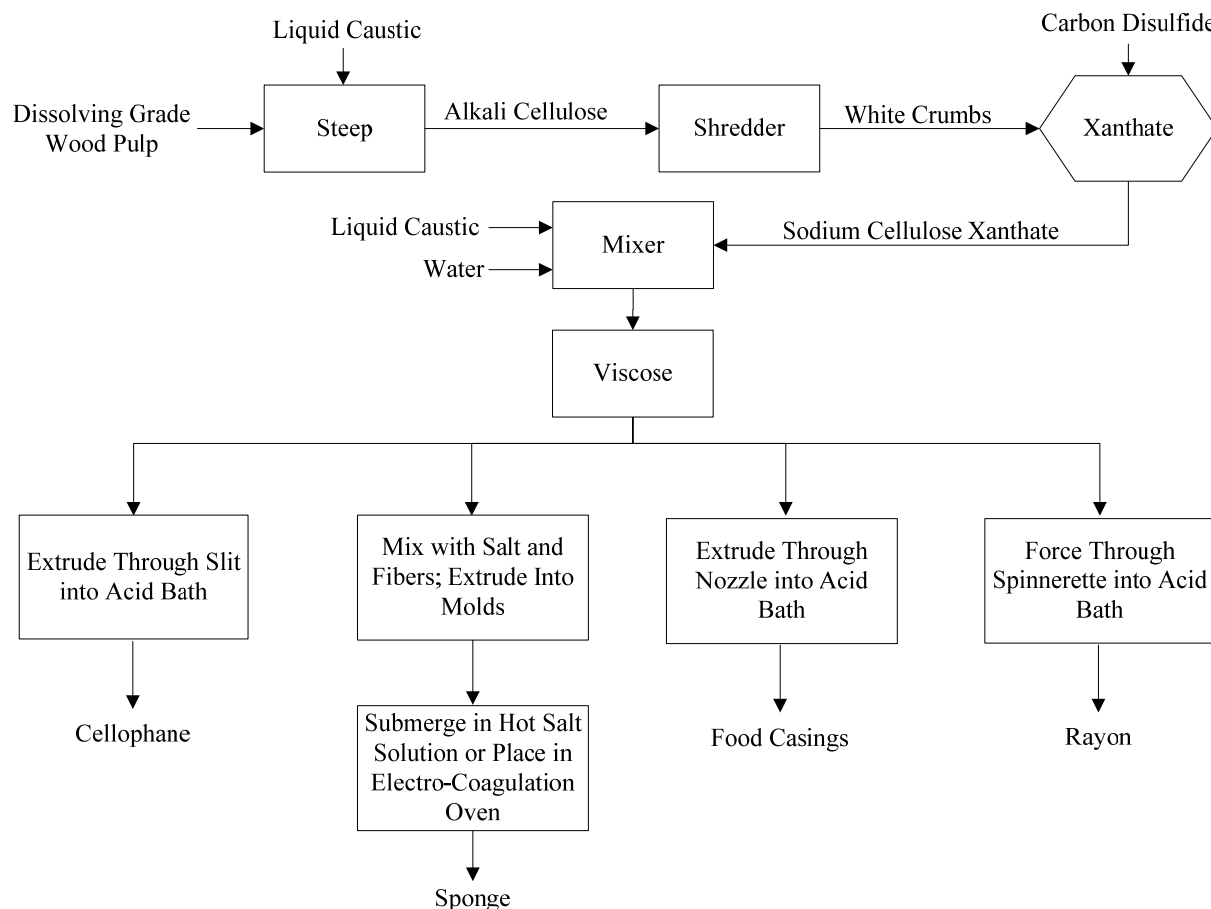


Figure 1. Simplified Process Flow Diagram for the Generic Viscose Process and Regenerated Cellulose Products

1.3 Background - Existing Effluent Guidelines and Regenerated Cellulose Manufacturing

The regenerated cellulose manufacturing process is closely related to two effluent limitation guidelines and standards (ELGs) point source categories: Organic Chemicals, Plastics, and Synthetic Fibers (OCPSF, 40 CFR Part 414) and Plastics Molding and Forming (PMF, 40 CFR Part 463). However, wastewater discharges from the manufacture of several cellulose products are not covered by any existing categorical ELGs or pretreatment standards.

1.3.1 *40 CFR Part 414*

The OCPSF ELGs (40 CFR Part 414, Subpart B) apply to wastewater discharged from the manufacture of rayon, a regenerated cellulose fiber. However, these regulations specifically exclude discharges from the manufacture of cellulose film, sponge, and meat casings (40 CFR §414.20). Further, these regulations do not include limitations for CS₂ and there are no rayon facilities currently operating in the United States. Appendix A includes further information on the applicability and subparts of 40 CFR Part 414.

1.3.2 40 CFR Part 463

The PMF industry includes facilities that are engaged in blending, molding, forming, or other types of processing of plastic materials. These processes commonly include extrusion, coating and laminating, thermoforming, calendaring, casting, foaming, cleaning, and finishing (U.S. EPA, 1984). EPA first promulgated ELGs for the PMF Category (40 CFR Part 463) on December 17, 1984 (49 FR 49040). There are three subcategories, all of which have best practicable control technology (BPT), new source performance standards (NSPS), pretreatment standards for existing sources (PSES), and pretreatment standards for new sources (PSNS) limitations.

The manufacture of cellulose products are not covered by Part 463 (the PMF Category). The product is made of regenerated cellulose using the viscose process, and Part 463 specifically excludes products manufactured from regenerated cellulose, as well as the molding and forming of regenerated cellulose (U.S. EPA, 1984).

40 CFR §463.1(g) Processes used to regenerate cellulose and to produce a product (e.g., rayon) from the regenerated cellulose are not subject to the effluent limitations guidelines and standards in this part. Processes that mold or form cellulose derivatives (e.g., cellulose acetate) are subject to the effluent limitations guidelines and standards in this part if they discharge process water.

The regenerated cellulose manufacturing process is similar to the operations regulated by 40 CFR Part 463 because water contacts extrusion equipment and regenerated cellulose products for cooling, cleaning, and finishing; however, regenerated celluloses are not plastic materials and are not regulated by the PMF ELGs. Table 1 describes the three subcategories regulated by the PMF ELGs.

Table 1. Applicability of Subcategories in the PMF Category

Subpart	Description	Applicability
A	Contact Cooling and Heating Water	Processes where water contacts plastic material or plastic products for the purpose of heat transfer.
B	Cleaning Water	Processes where water contacts the plastic products or shaping equipment for the purpose of cleaning.
C	Finishing Water	Processes where water contacts plastics products during finishing.

Source: *Preliminary Review of Prioritized Categories of Industrial Dischargers* (U.S. EPA, 2005a)

The Technical Development Document for Effluent Limitations Guidelines and New Source Performance Standards for the Plastics Molding and Forming Point Source Category (PMF TDD) provides the rationale for excluding regenerated cellulose manufacturers from the PMF ELGs. It states that plastic materials are a group of synthetic, organic materials composed of high molecular weight, long chain molecules. The definition of plastic materials in the PMF regulation also includes natural polymers that are combined with synthetic organic materials,

such as cellulose acetate. However, wholly natural organic materials, such as regenerated cellulose, are not included in the PMF definition of plastic materials.

The PMF TDD also states that the final step in the xanthate process used to regenerate cellulose is to wash the regenerated cellulose to remove dissolved salts and sulfur compounds within the product. Process water used in this step is not considered cleaning water as defined in the final PMF ELGs because it does more than just clean the surface of the regenerated cellulose.

Therefore, wastewater discharges from the manufacture of several cellulose products, including film, sponge, and food casings, are not covered by any existing categorical ELGs or pretreatment standards. Table 2 provides an overview of the cellulose products that are covered by existing ELGs and corresponding CS₂ limitations.

Table 2. Existing ELGs for Regenerated Cellulose Materials

Regenerated Cellulose Product	40 CFR Part 414 (OCPSF)	40 CFR Part 463 (PM&F)
Rayon ^a	Included, but no CS ₂ limits	Excluded
Cellulosic Film (Cellophane)	Excluded	Excluded
Cellulosic Sponge	Excluded	Excluded
Food Casing	Excluded	Excluded

Source: 40 CFR §414.20 and 40 CFR §463.1(g)

a – There are not rayon manufacturers currently operating in the United States.

1.3.3 Potential New Subcategories for the OCSPF Category

EPA reviewed the information provided in Tables 1 and 2 and determined that the cellulose products manufacturers using the viscose process to manufacture cellophane, cellulosic sponge, and food casings have processes, operations, wastewaters, and pollutants identical to the rayon manufacturers regulated by the OCPSF Category (Subpart B – Rayon Fibers). The end products at these facilities are all considered regenerated cellulose materials and are all produced using the same viscose process. For future screening level data review, EPA will classify the discharges from these facilities as a potential new subcategory of the OCPSF Category.

1.3.4 NPDES Permit Basis

Because wastewater discharges from the manufacture of several cellulose products, including film, sponge, and food casings are not covered by any existing categorical ELGs or pretreatment standards, permit writers have used state water quality standards, historical performance effluent data (HPED), other similar ELGs (i.e., OCSPF Subpart D – Thermoplastic Resins BPT), and best professional judgment (BPJ) as the basis for permit limits. The permits for wastewater discharges from cellulose products manufacturing include limits for the following pollutants: biological oxygen demand (BOD), total suspended solids (TSS), ammonia, ammonia nitrogen, total residual chlorines, oil and grease, and total organic carbon.

Only one NPDES permit (for Innovia Films, Tecumseh, KS, a cellulose products manufacturer), includes monitoring requirements for CS₂. Innovia Films is required to monitor CS₂ to avoid process upsets to the facility’s activated sludge treatment system. Other permitted facilities are not required to monitor CS₂, likely because there are no national water quality criteria for it, and other similar ELGs do not have limitations for CS₂.

1.4 Background – Industry Profile of U.S. Cellulose Manufacturers

EPA identified seven cellulose products manufacturers operating in the U.S. as of 2009. The final products of these facilities compete in markets with products made from alternative materials, especially plastics. Cellulose products have generally been declining in market share over time as newer non-cellulose products have been introduced (Beach, et al., 2000). The cellophane manufacturing industry has been declining since the 1950s due to new regulations and economic issues. The domestic market for rayon has also been eliminated because textile production has moved out of the United States (Beach, Houtven, Buckley, and Depro, 2000). Table 3 lists the operating U.S. cellulose products manufacturers and their discharges from the 2009 TRI and DMR databases, in pounds per year (LBY) and toxic-weighted pound equivalents (TWPE). Table 4 lists those manufacturing operations that appear to have been idled or closed prior to 2009.

Table 3. Operating U.S. Cellulose Products Manufacturers and Corresponding 2009 TRI and DMR Discharges

	Facility ^a	Location	Product Type	2009 TRI Data					2009 DMR Data		
				Discharge Type ^b	Basis of Estimate ^c	CS ₂ Pounds Released from Facility ^d	CS ₂ Pounds Released to Stream ^e	CS ₂ TWPE	Average Flow (MGD)	CS ₂ LBY	CS ₂ TWPE
1	3M Corporation	Elyria, OH	Cellulosic Sponges	Indirect	M2	NR	NR	NR	NA	NR	NR
2	3M Corporation	Tonawanda, NY	Cellulosic Sponges	Indirect	O	4,700	752	2,105	NA	NR	NR
3	Innovia Films Inc.	Tecumseh, KS	Cellophane	Direct	M2	26,500	26,500	74,300	2.22	24,100	53,100
4	Spontex Inc.	Columbia, TN	Cellulosic Sponges	Direct	M2	223	223	624	0.38	NR	NR
5	Viscofan USA Inc.	Danville, IL	Food Casings	Indirect	M2	14,000	2,240	6,270	NA	NR	NR
6	Viskase Corporation	Osceola, AR	Food Casings	Direct	O	473	473	1,320	1.16	NR	NR
7	Viskase Corporation	Loudon, TN	Food Casings	Indirect	M2	13,000	2,080	5,820	NA	NR	NR

Source: *Industry Profile of the Cellulose Products Manufacturing Facilities in the U.S.* (Schmidtke, 2000), *DMRLoads2009_v2*, *TRIRelases2009_v2*.

a – Cellulose manufacturing facilities that are currently operating in the United States.

b – ELGs control pollutant discharges at the point of discharges from industrial facilities and cover discharges directly to surface water (direct discharges) and discharges to POTWs (indirect discharges).

c – Basis of Estimate Descriptions: M1: continuous monitoring data or measurements; M2: periodic or random monitoring data or measurements; C: mass balance calculations, such as calculation of the amount of the toxic chemical in streams entering and leaving process equipment; E: published emission factors; E2: site-specific emission factors; and O: other approaches, such as engineering calculations.

d – Discharges include transfers to POTWs and do not account for POTW removals.

e – Discharges include transfers to POTWs and account for POTW removals.

NR – No data reported.

Table 4. Closed or Idled Cellulose Manufacturers in the United States

Facility Name	Location	Product Type	Facility Status in 2011	Type of Discharger ^a
Lenzing Fibers Corp.	Axis, AL	Rayon	Closed	Direct
Lenzing Fibers Corp.	Lowland, TN	Rayon	Closed	Direct
3M Corporation	Prairie du Chien, WI	Cellulosic Sponges	Idled	Indirect

Source: *Industry Profile of the Cellulose Products Manufacturing Facilities in the U.S.* (Schmidtke, 2000)

2.0 CARBON DISULFIDE PROPERTIES

All cellulose products manufacturers use CS₂ in the viscose process to regenerate cellulose. The CS₂ is used to react with aged alkali cellulose to form sodium cellulose xanthate. CS₂ is an extremely flammable and highly volatile chemical that is slightly soluble in water.

2.1 Chemical Properties of Carbon Disulfide

Table 5 presents chemical properties for CS₂ compared to acetone and ethanol. The Henry's Law Constant for CS₂ is 1,748 Pa m³/mol at 25° C (Love, 2011). Henry's Law Constant is the measure of the solubility of a gas in a liquid at a particular temperature, proportional to the pressure of that gas above the liquid (Kotz and Treichel, 1999). Chemicals with a higher Henry's Law Constant are more volatile. For example, CS₂ is highly volatile: the gas constant for CS₂ is approximately 403 times higher than that for acetone (4.02 Pa m³/mol).

Table 5. Chemical Properties of Carbon Disulfide, Acetone, and Ethanol

Chemical Properties	CS ₂	Acetone	Ethanol
Henry's Law Constant at 25°C	1,748 Pa m ³ /mol	4.02 Pa m ³ /mol	0.585 Pa m ³ /mol
Water Solubility	0.2% at 20° C	Soluble	Miscible
Evaporation Rate ^a	22.6	5.7	NA
National Fire Protection Association (NFPA) Flammability Rating ^b	4	3	3

Sources: EPA On-line Tools for Site Assessment Calculation; Fischer Scientific MSDS for Acetone and Ethanol; Love, 2011; Ohio EPA, 2010; and OSHA Guidelines for Carbon Disulfide.

a – The rate at which a material will vaporize when compared to the known standard rate of butyl acetate (evaporation rate = 1.0).

b – The NFPA flammability rating ranks the relative danger for a chemical. The higher the rating, the higher danger associated with the chemical flammability.

CS₂ is also hazardous to humans when inhaled. Table 6 presents the Occupational Safety & Health Administration (OSHA) and National Institute for Occupational Safety and Health (NIOSH) established permissible exposure limits (PELs) and recommended exposure limits (RELs) for CS₂, acetone, and ethanol. These exposure limits correspond to the amount or concentration of the substance in the air and may also contain skin designation. As part of the Occupation Safety and Health Guidelines, OSHA also established CS₂ PELs of 30 ppm (acceptable peak concentration for a 30 minute exposure) and 100 ppm (instantaneous maximum peak). NIOSH RELs also include a 10 ppm short-tem exposure limit.

Table 6. Inhalation Exposure Limits for Carbon Disulfide, Acetone, and Ethanol

Exposure Limits	CS ₂	Acetone	Ethanol
OSHA PELs (TWA) ^a	20 ppm ^b	1000 ppm	1000 ppm
NIOSH RELs (TWA) ^a	1 ppm	250 ppm	1000 ppm

Sources: Fischer Scientific MSDS for Acetone and Ethanol and OSHA Guidelines for Carbon Disulfide.

a – Time weighted average.

b – OSHA PEL for CS₂ is an 8-hour time weighted average (TWA) concentration.

Because CS₂ is highly volatile and flammable, facilities take additional precautions to reduce emissions to the air and sparks during the transfer of CS₂. These precautions include submerging the pipelines from storage tanks to the process in water trenches, transporting recovered CS₂ underground from the process to storage tanks, and transferring CS₂ by gravity or magnetically sealed pumps.

EPA contacted the Carbon Disulfide Coalition to determine its toxicity levels and fate and transport in water. The Akzo Nobel contact, a Carbon Disulfide Coalition member and a manufacturer of CS₂, provided the following freshwater toxicity levels:

- Freshwater, acute: 3 mg/L; and
- Freshwater, chronic: 1 mg/L.

The Akzo Nobel contact also stated that CS₂ volatilizes quickly from water. Historically, Carbon Disulfide Coalition scientists had difficulty measuring the solubility of CS₂, due to the rapid volatilization of free CS₂ into the vapor space. They found that the dissolved levels quickly dropped during the experiment. The Coalition concluded that the fate and transport of CS₂ in water would be volatilization, i.e., none would stay in solution (Love, 2011).

Table 7 presents the total air emissions and water discharges (to surface water or POTW) for the seven cellulose products manufacturers. The reported data confirm that the majority of the CS₂ evaporates, which corresponds with the physical/chemical properties. However, the estimated concentration of CS₂ in the water discharged from four facilities exceeds the CS₂ solubility at 25° C (0.2 %) (Love, 2011).

Table 7. TRI 2009 Carbon Disulfide Air Emissions and Water Discharges for Cellulose Products Manufacturers

	Facility	Location	Discharge Type ^a	Water Releases % of Total CS ₂ Discharges	Total CS ₂ Air Emissions	CS ₂ Discharge before POTW Removals
1	3M Corporation	Elyria, OH	Indirect	NA	187,000	NR
2	3M Corporation	Tonawanda, NY	Indirect	1.22 % ^b	379,000	4,700
3	Innovia Films Inc.	Tecumseh, KS	Direct	3.1 % ^b	820,000	26,500
4	Spontex Inc.	Columbia, TN	Direct	0.03 %	618,000	223
5	Viscofan USA Inc.	Danville, IL	Indirect	0.41 % ^b	3,360,000	14,000
6	Viskase Corporation	Osceola, AR	Direct	0.05 %	924,000	473
7	Viskase Corporation	Loudon, TN	Indirect	0.68 % ^b	1,890,000	13,000

Source: *TRIRelases2009_v2*.

a – ELGs control pollutant discharges at the point of discharges from industrial facilities and include discharges directly to surface water (direct discharges) and discharges to POTWs (indirect discharges).

b – The estimated CS₂ concentrations discharged to water from these facilities exceeds the CS₂ solubility at 25° C (0.2 %)

NR – No data reported.

NA – Not applicable.

2.2 Carbon Disulfide - Potential Pathways into the Environment

Releases of CS₂ from manufacturing facilities are almost exclusively to the atmosphere (ATSDR, 1996). However, facilities transfer CS₂ to wastewater during feedstock unloading and storage and air pollution control. Other than cellulose products manufacturing, industries that use CS₂ as a raw material or form it as a by-product include the manufacturing of soil disinfectant, development restrainer for instant color photography, aging of roasting coffee, pesticide intermediates, degreasing, chemical analysis, electroplating of gold and nickel, oil extraction, and dry cleaning (ATSDR, 1996). Natural sources of CS₂ in the environment include anaerobic ocean floors, wetlands, microbial activity in solids, certain crop plants and trees, volcano and marsh emissions, and vapor space above liquid sulfur (ATSDR, 1996).

3.0 WASTEWATER SOURCES

At cellulose products manufacturing facilities, the primary sources of wastewater containing CS₂ are railcar unloading, storage, viscose process steps, and air pollution control (Schmidtke, 2000). After reviewing the available data, the majority of the CS₂ enters the wastewater from the viscose processes used to regenerate the cellulose (KDHE, 2005; Martin, 2011).

3.1 Unloading and Storage

Most cellulose products manufacturing facilities receive CS₂ in the liquid or gas form by railcar. Because CS₂ is highly flammable, facilities unload CS₂ by water and/or nitrogen displacement to prevent fire or explosion. Water displacement is a method of unloading liquid CS₂ from railcars where water enters the railcar at the same flow rate that the CS₂ is expelled to the storage container. Nitrogen displacement is the same process; however, the inert gas fills the railcar as CS₂ is expelled. Facilities use water and/or nitrogen displacement to prevent

flammable, gaseous CS₂ emissions to the atmosphere and to preclude contact with oxygen (§40 CFR 63.5610).

Once in the storage pool and/or tank, water or inert gas is again used to contain the CS₂. If nitrogen is used, a nitrogen blanket is applied with enough pressure to keep the CS₂ in liquid form. If water is used, the liquid CS₂ is more dense than water, and a water blanket will also keep the CS₂ in liquid form. The liquid CS₂ remains at the bottom and is recovered or discharged to the wastewater treatment system. CS₂ does not evaporate from the unloading/storage system because the systems are closed to the atmosphere and are not mixed/aerated, the process that allows CS₂ to volatilize. Figure 2 shows the CS₂ unloading and storage processes by displacement.

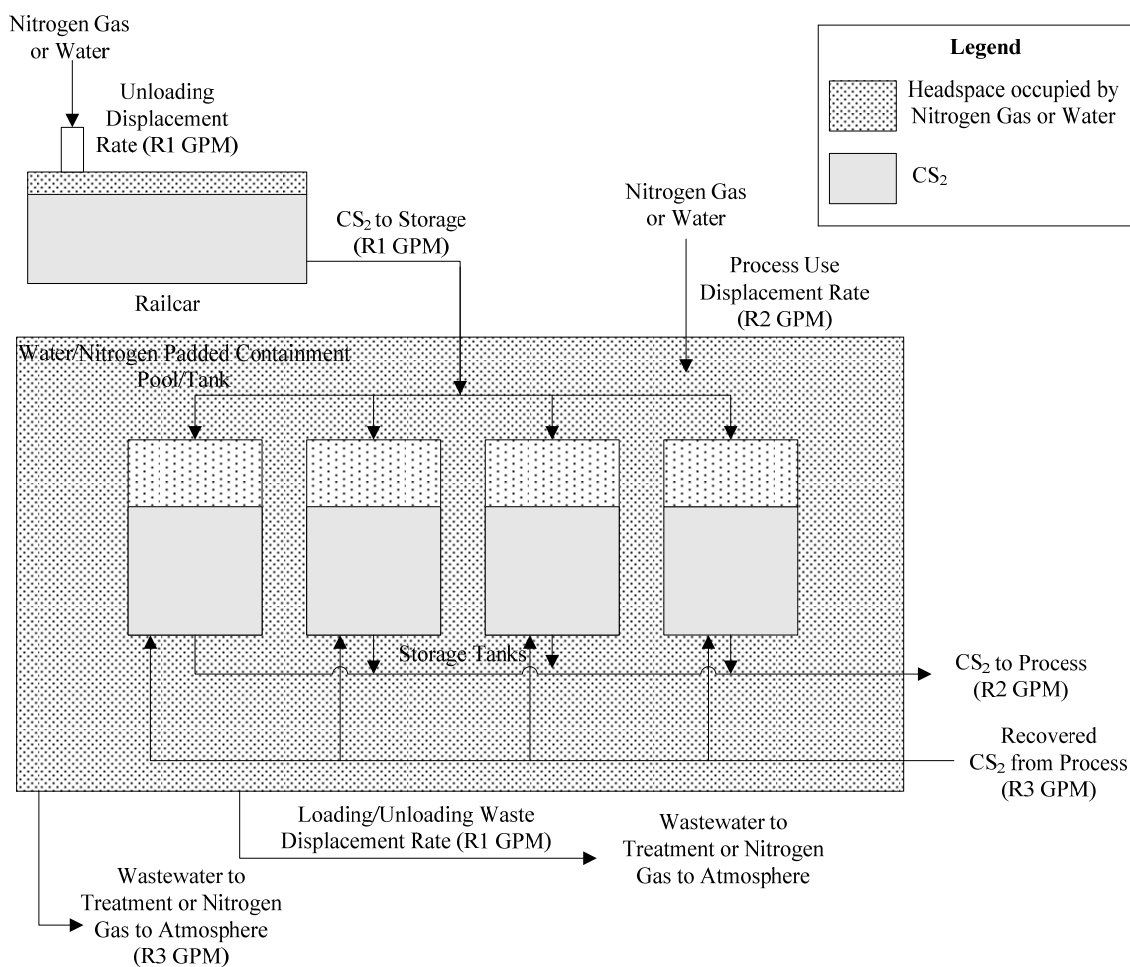


Figure 2. CS₂ Unloading and Storage System Using Containment Pool/Tank

Facilities using water displacement for raw material unloading generate CS₂-saturated wastewater during railcar unloading; water displaced from the CS₂ storage tank is sent to the facility's wastewater treatment system (Schmidtke, 2000).

After unloading CS₂, facilities store pressurized, liquid CS₂ in large tanks. Because CS₂ is extremely flammable, the facilities store the tanks with one or a combination of the following methods:

- Submerged under water in a containment pool;
- Raised above a water containment pool; or
- Covered by a nitrogen blanketing system.

Because liquid CS₂ has a higher specific gravity than water, any CS₂ that leaks during storage will settle to the bottom of the containment pool that a storage tank is submerged in or stored above.

In addition to underwater or nitrogen blanket storage, the facilities use a water or nitrogen padding system to fill headspace in the tank and further prevent contact with oxygen. The water or nitrogen padding is displaced into the water pool as CS₂ is loaded into the storage vessel. Conversely, the water or nitrogen padding in the storage tank fills the headspace as CS₂ is expelled for use in the manufacturing process, also shown in Figure 2.

Any water that is displaced from the unloading or storage in the pool and water padding is sent to the wastewater treatment system (Schmidtke, 2000). Table 8 presents the unloading and storage practices for each cellulose products manufacturing facility.

Table 8. Facility Carbon Disulfide Unloading and Storage Practices as of 2000

Facility		Facility Location	Facility Unloading Procedures		Facility Storage Procedures	
			Unloading System	Water Displacement ^a	Tank Location	Padding System
1	3M Corporation	Elyria, OH	N ₂	NA	Submerged in Water Pool	Water
2	3M Corporation	Tonawanda, NY	N ₂	Yes	Mounted Over Water Dike	Water
3	Innovia Films Inc. ^b	Tecumseh, KS	N ₂	No	Mounted Over Water Dike	N ₂
4	Spontex Inc.	Columbia, TN	Water	NA	NA	Water
5	Viscofan USA Inc.	Danville, IL	N ₂	No	Submerged in Bottom Sloped Water Pool	Water
6	Viskase Corporation	Osceola, AR	N ₂	NA	Submerged in Water Pool	N ₂
7	Viskase Corporation	Loudon, TN	N ₂	Yes	Submerged in Water Pool	N ₂

Source: Schmidtke, 2000

a – Some facilities use nitrogen to displace CS₂ from the railcar into storage tanks, but use a water dike or pool for the storage tank. Therefore, they create a wastewater stream as CS₂ enters the storage tank.

b – Innovia Films is the only facility where the CS₂ is not directly in contact with water as part of the unloading or storage processes at the facility. The facility contact confirmed that the majority of the facility’s discharges are from the viscose processes and CS₂ recovery at the plant.

3.2 Process Steps

CS₂ is combined with aged alkali cellulose to form sodium cellulose xanthate. After this reaction, the sodium cellulose xanthate is combined with liquid caustic and water in a coagulation bath. In order to control the sodium sulfate levels of the coagulation bath, water is continuously drained from this process step. Because the sodium cellulose xanthate contains some CS₂ from previous process steps, CS₂ is discharged with the wastewater (Martin, 2011).

Any wastewater overflow from the viscose solution steps also contains CS₂ discharges (Schmidtke, 2000). At this time, EPA is unable to determine what causes overflow from the viscose solution steps or the amount of wastewater that results from process overflow.

At one facility, CS₂ used in the viscose process is recovered through condensation. The facility uses steam to condense the CS₂, which is ultimately sent to the recovery plant. The CS₂ not captured from condensation is oil-scrubbed and recovered. Because some CS₂ is soluble in water, the steam condensate may contain CS₂. The steam condensate is sent to the facility's wastewater treatment plant (Martin, 2011).

3.3 Air Pollution Control

On June 11, 2002, EPA promulgated a NESHAP for Cellulose Products Manufacturing (40 CFR Part 63, Subpart UUUU, 67 FR 40055). The Cellulose Products Manufacturing NESHAP regulates the Miscellaneous Viscose Processes and Cellulose Ethers Production Categories. The Miscellaneous Viscose Process category includes cellulose food casings, rayon, cellulosic sponge, and cellophane manufacturing facilities. The NESHAP established emissions limits for hazardous air pollutants (HAPs), such as CS₂, carbonyl sulfide, ethylene oxide, methanol, methyl chloride, propylene oxide, and toluene. The resulting additional air pollution control may be transferring more CS₂ to the water via scrubbers (U.S. EPA, 2006).

Gaseous by-products formed during the regeneration of cellulose, including hydrogen sulfide and CS₂, are off-gassed from the process equipment. Facilities control emissions of these toxic gases using either a wet gas scrubber, a direct-contact condenser, or a biofilter system. In a wet gas scrubber, the air pollutants are absorbed by an aqueous solution. The wet scrubber removal efficiency for CS₂ varies depending on facility specific operations. For example, some systems can remove up to 65 percent of CS₂ prior to the stack (Marshall, 1998). The majority of the scrubbed CS₂ is either recovered or sent to wastewater treatment as scrubber effluent wastewater. At other facilities, CS₂ removal is low but the scrubber effluent wastewater may contain some CS₂ (Schmidtke, 2000).

In direct-contact condensers, water vapors are condensed out of the vent stream. The condensed water is sent to the wastewater treatment system. The remaining CS₂ vapors are pulled by a vacuum jet to a second direct-contact condenser. The CS₂ condenses because of the decrease in temperature and is sent to a settling tank with other process by-products. The condensed CS₂ is piped underground to the storage tanks in the water containment pool. The CS₂ emission reduction for these condenser systems is 99 percent (Schmidtke, 1998a).

Biofilters are also used as air pollution control at cellulose products manufacturing facilities. Each biofilter (bed) has a plenum at the bottom (air space), a midsection that contains grating and media that contain microbes, and a cover on top. The gases enter the bottom of the bed and exit from the top. The removal efficiency of the biofilter varies based on process conditions; however, normal CS₂ reductions across the bed are approximately 80 percent (Nicholson, 2000). Wastewater from the biofilter, which may contain some CS₂, is sent to wastewater treatment facilities. The spent media from the biofilter are neutralized with lime and hauled offsite (Nicholson, 2000).

4.0 REGENERATED CELLULOSE PROCESS WASTEWATER TREATMENT

Table 9 summarizes the wastewater treatment used by the three cellulose products manufacturers where EPA obtained data (Schmidtke, 2000). Table 10 summarizes the possible sources of wastewater containing CS₂ and the methods of wastewater treatment specific to each plant.

Table 9. Cellulose Products Wastewater Treatment

Product	Pretreatment Used by Indirect Dischargers	Treatment Used by Direct Dischargers
Cellophane	NA	Neutralization, settling, equalization, second neutralization, aeration, and clarification (i.e., activated sludge)
Food Casings	Neutralization, filtration and settling. Achieved CS ₂ concentrations of 5-20 parts per million (ppm).	Neutralization using lime, equalization, and clarification.
Cellulosic Sponges	Neutralization and oxidization	Equalization, aeration, and clarification (activated sludge).

Source: *Industry Profile of the Cellulose Products Manufacturing Facilities in the U.S.* (Schmidtke, 2000).
 NA – Not applicable.

In addition to wastewater treatment, facility management practices also affect how much CS₂ is discharged in wastewater. For example, the Loudoun, TN Viskase facility formerly used a water blanket during CS₂ unloading. This led to 10,000 gallons of CS₂ saturated water being discharged to the wastewater treatment facility every time CS₂ was unloaded. By converting to the nitrogen blanket system, the Viskase facility eliminated a significant source of CS₂ wastewater discharges (Schmidtke, 1998b).

Table 10. Confirmed Sources of Wastewater and Wastewater Treatment at Cellulose Manufacturing Facilities

Facility Name	Location	Product Type	Possible Sources of Wastewater	Wastewater Treatment	Type of Discharger ^a
1 3M Corporation	Elyria, OH	Cellulosic Sponges	- Biofilter	- Oxidation - Neutralization - Holding tanks	Indirect
2 3M Corporation	Tonawanda, NY	Cellulosic Sponges	- Storage tank displaced water - Rotary vacuum filters	- Neutralization	Indirect
3 Innovia Films Inc.	Tecumseh, KS	Cellophane	- Viscose steps - Extrusion/regeneration	- Neutralization - Settling basin - Equalization basin - Activated Sludge	Direct
4 Spontex Inc.	Columbia, TN	Cellulosic Sponges	- Storage tank displaced water - Railcar blowoff - H ₂ S scrubber vacuum pump discharge	- Equalization - Activated Sludge	Direct
5 Viscofan USA Inc.	Danville, IL	Food Casings	- Overflow of storage tank submersion water - Viscose steps (slurry, ripening, filtration) - Extrusion/regeneration - Washing - Acid systems (acid recovery, basement systems, anhydrous department)	- Mixing basin - Neutralization - Settling basins - Discharge basin (stack)	Indirect
6 Viskase Corporation	Osceola, AR	Food Casings	- Viscose steps (ripening room trenches) - Extrusion/regeneration (extrusion wash water, extrusion acid) - Deaerator condensate - Waste handling	- Neutralization (lime) - Equalization - Clarifiers	Direct
7 Viskase Corporation	Loudon, TN	Food Casings	- Extrusion/regeneration - Washing	- Filter - Neutralization	Indirect

Source: *Industry Profile of the Cellulose Products Manufacturing Facilities in the U.S.* (Schmidtke, 2000).

a – ELGs control pollutant discharges at the point of discharges from industrial facilities and cover discharges directly to surface water (direct discharges) and discharges to POTWs (indirect discharges).

5.0 FACILITY DISCHARGE DATA

EPA reviewed all available CS₂ concentration data for facilities presented in Table 3. EPA located wastewater treatment information for two facilities: Innovia Films, Inc in Tecumseh, KS and Viskase Corporation in Loudon, TN.

5.1 Innovia Films, Inc in Tecumseh, KS

Innovia Films, Inc in Tecumseh, KS manufactures cellophane used primarily in food packaging. EPA obtained 2009 discharge monitoring report (DMR) data for this facility, the only cellulose products manufacturing facility required to monitor for CS₂. EPA obtained facility-specific waste stream and wastewater treatment data from the facility's permit and fact sheet.

5.1.1 Waste Streams

Acid, alkaline, and neutral waste streams are generated from the cellulose production process. All process wastewaters are commingled, treated, and discharged through the treatment plant discharge (outfall 001). Approximately 2.0 MGD wastewater is treated and discharged through Outfall 001. Table 11 describes the facility waste streams and typical flow.

Table 11. Innovia Films, Inc Waste Streams

Waste Stream	Description	Typical Flow (MGD)
Alkaline	Domestic waste flows and alkaline waste streams from the mix house, finishing, coating, casting, and the viscose manufacturing areas (VMA)	0.67
Acid	Acid waste process flows from casting areas and the powerhouse ^a	0.6
Neutral	Condensate flow from the hot well area	0.68

Source: Facility Fact Sheet (KDHE, 2005).

a – The powerhouse discharge also includes water from reverse osmosis cleaning and demineralizer washes.

CS₂ enters the wastewater from different processes at the plant (Martin, 2011):

- Start up procedures that call for the dumping of liquid viscose (alkaline waste stream);
- Overflow from the viscose process, which results in various losses of CS₂ as liquid viscose (alkaline waste stream);
- Continuous dumping of the coagulation bath solution to the wastewater treatment system, which is dumped to control the balance of sodium sulfate in the viscose process (alkaline waste stream);
- Casting area water and wash water (acid waste stream); and
- CS₂ recovery system (neutral waste stream), which is described below.

Because CS₂ is volatile valuable feedstock, the facility recovers volatilized CS₂ used in the process in the CS₂ recovery system (Martin, 2011). The following process steps result in excess or liberated CS₂ air emissions:

- The xanthation step in the viscose process, where CS₂ is sprayed on alkali cellulose to produce xanthate (an intermediate product in the cellophane process);
- The extrusion/casting step, where the viscose solution is soaked in concentrated and dilute sulfuric acid baths, which liberates CS₂ and hydrogen disulfide; and
- The extrusion/casting step where the cellulose is washed in a hot water bath, which also liberates CS₂.

At the CS₂ recovery plant, the facility uses steam to recover the CS₂. Although the majority of the CS₂ is recovered, the facility contact stated that some CS₂ is captured in the steam condensate which is transferred to the wastewater treatment system (Martin, 2011).

5.1.2 Wastewater Treatment

The plant uses an activated sludge extended aeration process to treat the wastewater. The acid wastestream is first neutralized using hydrated lime in settling pond #1. The neutralized acid stream is then commingled with the alkaline and neutral wastestreams in settling pond #2. The combined wastestream is pumped to the equalization basin. As the wastestream is pumped from the equalization basin to the activated sludge aeration basin, the plant adds phosphoric acid and ammonium hydroxide to provide supplemental nutrients for biological treatment. The excess activated sludge is settled in clarifiers, and the treated effluent is discharged to the Kansas River through a long outfall sewer. The sludge is concentrated by a centrifuge and land applied (KDHE, 2005). Figure 3 diagrams the Innovia Films wastewater treatment system (Mester, 2011).

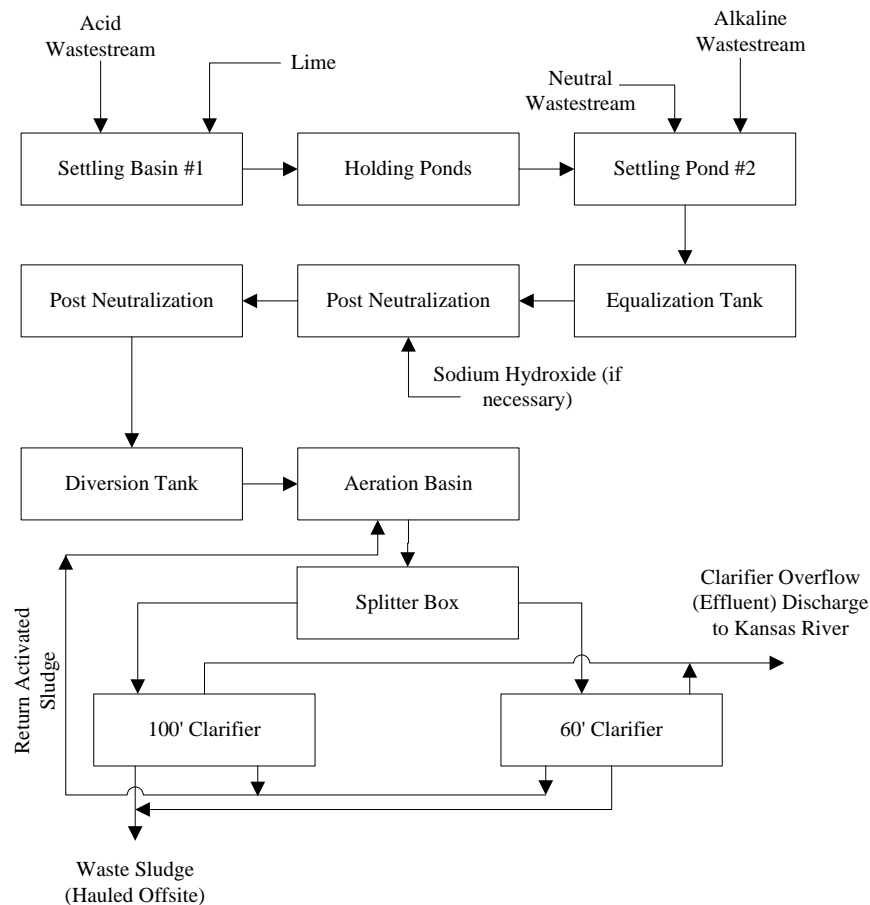


Figure 3. Block Diagram of Innovia Films Wastewater Treatment System

5.1.3 Discharge Data

Innovia Films’ permit requires the facility to monitor for CS₂ because concentrations at 35 mg/L or greater inhibit the activated sludge process. The permit requires remedial action if the CS₂ concentration exceeds 17.5 mg/L. The facility analyzes the CS₂ samples using EPA Method 624.

The facility samples for CS₂ at the splitter box, which receives flow from the aeration basin and feeds the flow to the clarifiers. The overflow (treated effluent) from the clarifiers is piped underground by gravity to the outfall. The outfall discharges to the receiving stream approximately 7,000 feet from the splitter box. Table 12 shows the 2008, 2009, and 2010 CS₂ concentration data for Innovia Films, Inc.

Table 12. 2008, 2009, and 2010 Carbon Disulfide Concentration Data for Innovia Films, Inc.

Outfall	Date	Monthly Minimum Concentration (mg/L)	Monthly Average Concentration (mg/L)	Monthly Maximum Concentration (mg/L)
001	31-Jan-08	0.63	2.03	2.86
001	29-Feb-08	1.58	4.98	8.45

Table 12. 2008, 2009, and 2010 Carbon Disulfide Concentration Data for Innovia Films, Inc.

Outfall	Date	Monthly Minimum Concentration (mg/L)	Monthly Average Concentration (mg/L)	Monthly Maximum Concentration (mg/L)
001	31-Mar-08	7.11	9.48	12.5
001	30-Apr-08	0.56	0.56	0.56
001	31-May-08	0.52	0.74	0.98
001	30-Jun-08	0.13	0.55	0.99
001	31-Jul-08	0.26	0.36	0.56
001	31-Aug-08	0.75	0.75	0.75
001	30-Sep-08	3.23	5.76	8.29
001	31-Oct-08	0	0	0
001	30-Nov-08	3.51	8.34	13.1
001	31-Dec-08	0	2.14	4.29
001	31-Jan-09	6.02	9.77	17.1
001	28-Feb-09	5.19	5.19	5.19
001	31-Mar-09	6.63	10.67	14.5
001	30-Apr-09	0.25	0.39	0.52
001	31-May-09	0.21	0.66	1.53
001	30-Jun-09	0.07	0.22	0.33
001	31-Jul-09	0.06	0.86	1.67
001	31-Aug-09	0.25	0.25	0.25
001	30-Sep-09	0.1	0.15	0.21
001	31-Oct-09	0.16	0.85	1.55
001	30-Nov-09	0	0.12	0.25
001	31-Dec-09	4.14	4.14	4.14
001	31-Jan-10	1.18	2.79	4.62
001	28-Feb-10	1.74	3.22	4.7
001	31-Mar-10	2.42	2.42	2.42
001	30-Apr-10	0.88	1.54	2.14
001	31-May-10	2.84	2.84	2.84
001	30-Jun-10	0	0.07	0.14
001	31-Jul-10	0.28	0.42	0.55
001	31-Aug-10	0.24	0.27	0.30
001	30-Sep-10	0.88	0.88	0.88
001	31-Oct-10	0.79	0.84	0.88
001	30-Nov-10	0.52	1.10	1.79
001	31-Dec-10	2.94	3.50	4.07

Source: Envirofacts

As shown in Table 12, a total of 10 average concentrations (28 percent) exceed 3 mg/L, the acute freshwater toxicity level for CS₂, and a total of 17 average concentrations (47 percent) exceed 1 mg/L, the chronic freshwater toxicity level for CS₂ (Love, 2011). There are no concentrations above the 17.5 mg/L permit monitoring concentration. The Carbon Disulfide Coalition finds these concentrations are inconsistent with their experiences. The coalition reviewed analytical values from a wastewater treatment system consisting of aeration and neutralization only. The concentrations of CS₂ at the discharge points were less than 1 mg/L, with an overall CS₂ removal over 99 percent (Love, 2011). The final effluent concentrations are likely less than those taken at the splitter box, because after the sampling point, the wastewater goes through clarification and travels over 1.3 miles through a pipe to the surface water, allowing for further volatilization.

5.2 Viskase Corporation in Loudon, TN

The Viskase Corporation in Loudon, TN is a food casings manufacturer, one of two operated by Viskase Corporation, the world's largest producer of small-sized food casings (Schmidtke, 1998b).

5.2.1 *Wastewater Source and Treatment*

The Viskase Corporation plant uses approximately 1.95 MGD of water. Wastewater from the extrusion, regeneration, and washing areas is sent to the wastewater treatment facility. At the wastewater treatment plant, coarse materials are removed from the waste stream and the wastewater is neutralized with lime. The wastewater is sent off site for treatment at a POTW. The CS₂ levels in the wastewater sent to the POTW typically vary between 5 and 20 ppm (Schmidtke, 1998b).

5.2.2 *Discharge Data*

In 2009, EPA contacted Viskase Corporation and the local pretreatment coordinator to confirm CS₂ discharges. The facility's industrial user permit limit for CS₂ is 5 mg/L. Viskase collects samples at the point where the discharge leaves the facility and enters a pipe connected to the adjacent POTW. In addition, the POTW collects samples at the other end of this pipe, where the Viskase discharge enters the POTW. These samples are analyzed for CS₂. The combined data show that the facility releases close to 100,000 pounds of CS₂ to the POTW, but the POTW receives less than 3,000 pounds per year from the discharge pipe per year. This decrease in pollutant load suggests the possible volatilization of 97,000 pounds per year of CS₂ within the discharge pipe (Birkholtz, 2009; Glarrow, 2009; U.S. EPA, 2009).

The POTW provided CS₂ concentration measurements taken at the influent to their treatment system. Table 13 shows the results of these sampling events.

Table 13. Carbon Disulfide Concentration Data for Viskase Corporation in Loudon, TN

Date	POTW Measurement at Treatment Works Influent (mg/L)	Discharge Limit (mg/L)
13-Feb-06	12	5
14-Aug-06	<0.001	5
20-Feb-07	<0.001	5
19-Sept-07	1.1	5
1-Apr-08	1.4	5
19-Aug-08	<0.001	5
3-Feb-09	<0.001	5

Source: Facility Contact (Birkholz, 2009).

The CS₂ concentrations for Viskase Corporation do not exceed the discharge limit of 5 mg/L, except for the February 2006 concentration. More importantly, these discharges represent the CS₂ concentrations prior to treatment through the POTW. Therefore, the CS₂ concentrations are likely below levels of detection in the POTW effluent, and are likely of no concern to human health and aquatic life.

6.0 CONCLUSIONS

The conclusions of the cellulose products manufacture review are as follows:

- From available data, EPA identified seven active regenerated cellulose manufacturers in the U.S. These facilities use and discharge wastewater containing CS₂ generated from CS₂ storage, recovery, and the viscose process. EPA continues to resolve the overall question of how significant concentrations of CS₂ (ranging from values below detection to 17.1 mg/L) remain in the water after treatment, although CS₂ is highly volatile (430 times more volatile than acetone, for example).
- There are three direct dischargers, but only one facility collects data on CS₂ in their wastewater. This facility, Innovia Films, Inc, discharges up to 17.1 mg/L of CS₂ after activated sludge wastewater treatment. There are 17 of 36 monthly average concentrations in the facility's 2008 through 2010 CS₂ data that are above the chronic freshwater toxicity level of 1 mg/L. The concentration of CS₂ entering the receiving stream is likely lower, because the sample point is more than one mile from the final discharge point, allowing for further CS₂ volatilization in the discharge pipe. The measurements reported by Innovia Films are inconsistent with information received from the Carbon Disulfide Coalition. Based on Coalition solubility and volatility data, the CS₂ concentrations would be less than 1 mg/L following aeration.
- There are four indirect dischargers, but EPA obtained data from only one facility on CS₂ in their wastewater. Because facilities pre-treat their wastewater, and it is processed through a POTW, CS₂ concentrations for indirect dischargers likely are well below aquatic life criteria.
- The cellulose products manufacturers using the viscose process include a small number of U.S. facilities. For this reason, EPA considers these discharges are best controlled by facility-specific permitting assistance.

EPA prioritizes point source categories with existing regulations for potential revision based on the greatest estimated toxicity to human health and the environment, measured as TWPE. Based on the above conclusions, EPA will categorize these discharges with Part 414, OCPSF for future annual reviews. EPA is assigning this category with a lower priority for revision (i.e., this category is marked with "(3)" in the "Findings" column in Table V-1 in the Federal Register notice that presents the 2011 annual review of existing effluent guidelines and pretreatment standards).

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**APPENDIX A
40 CFR Part 414**

EPA first promulgated ELGs for the OCPSF Category (40 CFR Part 414) on November 5, 1987 (52 FR 42568). This category consists of seven subcategories that apply to the manufacture of products and product groups, as shown in Table A-1 with corresponding Standard Industrial Classification (SIC) codes and applicability. Subparts B through H have limitations for biochemical oxygen demand (BOD₅), total suspended solids (TSS), and pH. The regulation also includes limitations and/or pretreatment standards for certain toxic pollutants in three additional subparts:

- Subpart I — Direct Discharge Point Sources That Use End-of-Pipe Biological Treatment;
- Subpart J — Direct Discharge Point Sources That Do Not Use End-of-Pipe Biological Treatment; and
- Subpart K — Indirect Discharge Point Sources.

Table A-1. Applicability of Subcategories in the OCPSF Category

Subpart	Subcategory Title	Corresponding SIC Code(s)^a	Subcategory Applicability
B	Rayon Fibers	2823: Cellulosic Manmade Fibers	Cellulosic manmade fiber (Rayon) manufactured by the Viscose process.
C	Other Fibers	2824: Synthetic Organic Fibers, Except Cellulosic	All other synthetic fibers (except Rayon) including, but not limited to, products listed in Section 414.30.
D	Thermoplastic Resins	28213: Thermoplastic Resins	Any plastic product classified as a thermoplastic resin including, but not limited to, products listed in Section 414.40.
E	Thermosetting Resins	28214: Thermosetting Resins	Any plastic product classified as a thermosetting resin including, but not limited to, products listed in Section 414.50.
F	Commodity Organic Chemicals	2865: Cyclic Crudes and Intermediates, Dyes and Organic Pigments 2869: Industrial Organic Chemicals, NEC	Commodity organic chemicals and commodity organic chemical groups including, but not limited to, products listed in Section 414.60.
G	Bulk Organic Chemicals	2865: Cyclic Crudes and Intermediates, Dyes and Organic Pigments 2869: Industrial Organic Chemicals, NEC	Bulk organic chemicals and bulk organic chemical groups including, but not limited to, products listed in Section 414.70.
H	Specialty Organic Chemicals	2865: Cyclic Crudes and Intermediates, Dyes and Organic Pigments 2869: Industrial Organic Chemicals, NEC	All other organic chemicals and organic chemical groups including, but not limited to, products listed in the OCPSF Development Document (Vol. II, Appendix II-A, Table VII).

Source: *Product and Product Group Discharges Subject to Effluent Limitations and Standards for the Organic Chemicals, Plastics, and Synthetic Fibers Point Source Category — 40 CFR 414, Table 2-2* (U.S. EPA, 2005b).

a – During the 2009 annual review EPA developed a crosswalk between SIC codes and NAICS codes. Because there is not a direct match EPA did not report NAICS codes.

NEC – Not elsewhere classified.