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Evaluation of Ammonium and Iodine Decontamination Factors for Hanford's Waste Treatment and Immobilization Plant Feed in the Effluent Treatment Facility

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) reviewed the unit operations in the Effluent Treatment Facility (ETF) at the Hanford site to estimate the partitioning of iodine and ammonia when processing Waste Treatments and Immobilization Plant (WTP) feed. The evaluation consisted of literature and vendor data reviews and no experiments were performed. A list of the unit operations reviewed, along with estimated decontamination factors (DFs) for both iodine and ammonia are shown in the table below. With the exception of the Peroxide Decomposer, reasonable estimations of the decontamination factors for all operations are provided.

Estimated Decontamination Factors of Hanford ETF Unit Operations.

Unit Operation	Ammonium DF	Iodine DF
Filtration	1	1
UV Oxidation	1	1
Peroxide Decomposer	1	Unknown*
Degas Column	1	1
Reverse Osmosis	5.4	33
Evaporation	> 20	≈ 5
Ion Exchange	> 100	> 100
Off-Gas System	1	1

* Depending on the pH, some iodine removal may occur in this unit operation.

Adsorption of iodine species by the bed material in the peroxide decomposer, whether activated carbon or a replacement media, could not be determined from the literature data. It is likely that some adsorption will occur, but the literature data is not definitive enough to allow a DF to be estimated.

Based on this information (assuming that bulk iodine is not removed in the peroxide decomposer), the bulk of the iodine and ammonia species are expected to be removed from the ETF waste stream in the reverse osmosis reject solution which is concentrated in the evaporator and discharged to a secondary waste stream. Approximately 20% of the iodine and less than 5% of the ammonia will be cycled back from the reverse osmosis process through the surge tank with the incoming ETF feed. Subsequent ion exchange treatment of the ETF process stream is expected to remove the remaining minor amounts of iodine and ammonia species which are transferred in the eluate from the columns to the evaporator.

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LIST OF ABBREVIATIONS

DF	Decontamination Factor
DFLAW	Direct-Feed Low Activity Waste
ETF	Effluent Treatment Facility
HEPA	High-Efficiency Particulate Air
HOI	Hypoiodous Acid
IX	Ion Exchange
LERF	Liquid Effluent Retention Facility
RO	Reverse Osmosis
SALDS	State-Approved Land Disposal Site
SRNL	Savannah River National Laboratory
SRWT	Secondary Waste Receiving Tank
UV	Ultraviolet
WRPS	Washington River Protection Solutions
WTP	Waste Treatment and Immobilization Plant

1.0 Introduction

Savannah River National Laboratory (SRNL) reviewed the Effluent Treatment Facility (ETF) at the Hanford site to determine estimated partitioning of iodine and ammonium when processing Waste Treatment and Immobilization Plant (WTP) feed. The review consisted of literature and vendor data reviews; no experimentation was performed. If an estimated decontamination factor (DF) cannot be determined from the available data, then the gap in available data is noted. A simplified block diagram of the ETF is provided in Figure 1-1 that shows principal operations in the facility expected for the Direct-Feed Low Activity Waste (DFLAW) mission.

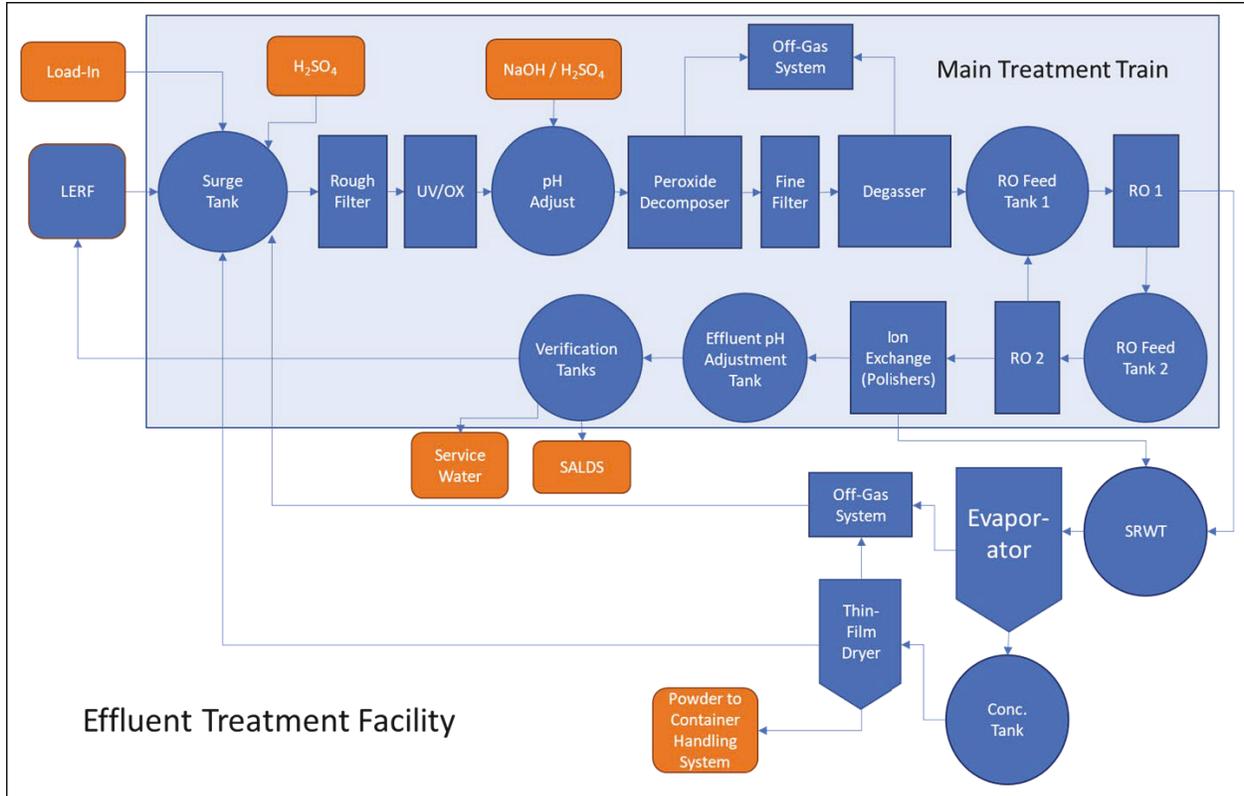


Figure 1-1. Simplified Diagram of Hanford's Effluent Treatment Facility

The partitioning of iodine and ammonium was evaluated for each unit operation listed below:

- Filtration
- Ultraviolet (UV) Oxidation
- Peroxide Decomposer
- Degas Column (Degasser)
- Reverse Osmosis (RO)
- Ion Exchange (IX)
- Evaporation of RO/IX reject
- ETF Offgas system

1.1 Estimated Form of Iodine and Ammonium in the ETF Feed

Any form of iodine that is fed to the Waste Treatment and Immobilization Plant (WTP) melter and partitions to an aqueous phase by the off-gas system will be present as iodide ion (I⁻) or iodate ion (IO₃⁻) at the anticipated pH near neutral, depending on the oxygen content of the stream.¹ The iodine is in the form of iodide ion or iodate ion in the influent to the ETF. As the pH is adjusted to ~5, some of the iodide/iodate can convert to iodine (I₂), although this is expected to be a minor amount because of the low concentration, which drives the iodine to convert to hypiodous acid (HIO). If iodine is present in the liquid waste at low concentrations as iodide ion, acidification can cause it to partially form molecular iodine (I₂) if hypiodous acid is formed, as shown in Equation (1) below.² This is similar to its behavior in surface waters.³



The majority of ammonia emissions are expected to come from the ETF Surge Tank vent prior to waste acidification. The feed within the Surge Tank is adjusted to pH 5-5.5 using sulfuric acid, converting volatile ammonia gas in solution to non-volatile ammonium sulfate. At this pH, ammonia is expected to be (> 99%) in the form of ammonium ion (NH₄⁺) (RPP-CALC-62964).

2.0 Results and Discussion

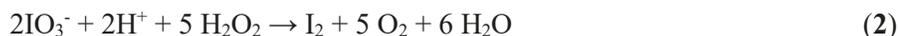
The fate of iodine and ammonium are evaluated for the select unit operation described below.

2.1 Filtration

No particulate species containing iodine or ammonium are expected to be formed during ETF processing. Therefore, no iodine or ammonium is expected to be removed by filtration (DF = 1).

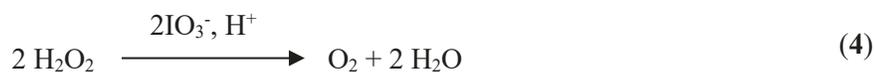
2.2 UV Oxidation

As discussed above, there are two forms of iodine that may be present in the feed to the organic destruction unit. Organics are destroyed in ETF by injecting 200^a mg/L of hydrogen peroxide into the waste water as it passes through one of four Calgon ultraviolet (UV)/oxidation chambers containing six lamps per chamber. The hydrogen peroxide produces two hydroxyl radicals (OH) upon ultraviolet radiation.⁴ Although iodine chemistry with strong oxidizers has been studied as part of an ozonation step,⁵ the ozone forms the same hydroxyl radical as the ultraviolet radiation with hydrogen peroxide, so the iodine would be expected to react to form the same products using hydrogen peroxide and UV. Although literature exists on iodine and hydrogen peroxide chemistry, the combination of peroxide and UV light makes the conditions more similar to those with ozone because of the hydroxyl radical formation. If the iodine is present in the liquid waste as iodate, it is not expected to react with hydrogen peroxide or the hydroxyl radicals since iodate is fully oxidized. Iodate is the product of the reaction of iodide with hydrogen peroxide, and is known to catalyze the acidic decomposition of hydrogen peroxide to water and oxygen in the Bray-Liebafsky reaction.⁶ Similarly, if iodine were present as other forms, such as molecular iodine, it would also react with the hydrogen peroxide to form iodate ions. This is also how it reacts in an intermediate step of the Bray-Liebafsky reaction.⁶ The reduction and oxidation reactions in the absence of UV light are shown in Equations (2) and (3) below, where it is shown that peroxide can also reduce iodate to form molecular iodine:



Overall, these two reactions result in the iodate-catalyzed oscillating peroxide decomposition referred to as the Bray-Liebafsky reaction shown in Equation (4):

^a Hydrogen peroxide addition is currently reduced due to downstream issues



If iodine is present in the liquid waste at low concentrations as molecular iodine in the absence of UV or hydrogen peroxide, it would initially react to form iodide and hypoiodous acid (HOI). This is similar to its behavior in surface waters and was discussed in the above section:³



The hypoiodous acid could then be oxidized to iodate ion, disproportionate to iodide and iodate ion (in the absence of hydrogen peroxide) or be reduced to iodide ion by further reaction with hydrogen peroxide. However, reduction to iodide is unlikely while the UV oxidation unit is operating due to the presence of hydroxyl radicals.³ Any dissolved iodine present can react with hydrogen peroxide to form iodate under these ultraviolet oxidizing conditions.⁶ It is also possible that the hypoiodous acid and iodine react with the organic chemicals present in the waste stream to form iodo-organics; although this is expected to have an overall minimal impact because the organics and any produced iodo-organics formed would be subsequently destroyed by the oxidizing conditions of the UV/hydrogen peroxide reaction. In the organic destruction unit, the hydroxyl radicals abstract a hydrogen ion from the organic chemicals whether or not they contain an iodine atom, producing a radical that further oxidizes, ultimately to carbon dioxide. Similarly, use of ozone to treat natural waters spiked with iodide (50 µg/L) was observed to convert >90% of the iodide to iodate ion, and iodo-trihalomethanes (e.g. iodoform) were absent.⁵ Although not demonstrated, the similar formation of hydroxyl radical in the UV/hydrogen peroxide system would be expected to result in the same products. The reaction rate of ozone oxidation of iodide to hypoiodous acid was shown to be $2\text{E}9 \text{ M}^{-1}\text{s}^{-1}$, and the subsequent ozone oxidation to iodate was measured to be $3.6\pm 1\text{E}4 \text{ M}^{-1}\text{s}^{-1}$ at pH <8.⁷ Although obvious differences would likely be observed with UV/hydrogen peroxide, the overwhelming amount of peroxide and UV irradiation versus small amounts of iodine would likely produce these same oxidized products at high rates. Other reactions may also occur, such as if nitrite ion is present in solution, iodide ion can react with it in acid to form iodine (I₂).⁸ If that were to occur, the subsequent reactions of iodine discussed above would then occur, and the result would be the same formation of iodide and hypoiodous ions. However, the concentration of nitrite is <1 mg/L and this reaction is not significant. Other reactions with ions in solution could also occur, but the dominant species are carbonate, oxalate, and nitrate. The molar concentrations of the iodine species are very low, and any secondary iodine byproducts are expected to undergo the same reactions discussed above. These species should partition in the subsequent processing steps in the ETF in the same way.

Although under some conditions, particularly without UV, iodate can also be reduced to iodine (I₂) by hydrogen peroxide,⁹ this is unlikely in the strongly oxidizing conditions caused by the UV irradiation. It is expected that the net effect is that the iodine will be predominantly in the form of soluble iodate in the effluent from the UV chambers. While this has not been proven with the UV/hydrogen peroxide system, it is expected to yield the same result as the analogous reaction with ozone at these very low concentrations of iodine species.

Reaction of hydrogen peroxide with ammonia is a strong function of pH. Although hydrogen peroxide and ammonia do not react in the dark, in the presence of UV light the hydrogen peroxide will generate peroxy free radicals, which can react with ammonia but not ammonium ion.¹⁰ The analogous oxidation of ammonia by peroxone (i.e., ozone and hydrogen peroxide without UV) can be accomplished at pH 8-11, but the ammonium ion dominates at lower pH and is not reactive. The same effect would be expected in the peroxide/UV system in the ETF. The product of the alkaline reaction would be nitrate ion. Similar observations of the photocatalytic decomposition of ammonia are found in studies of UV irradiation of ammonia and hydrogen peroxide. Below pH 8, the reaction to destroy ammonia and yield nitrate is negligible.¹¹ It is expected in the ETF that some reaction between hydrogen peroxide and ammonia could occur in the UV chambers to produce nitrite or nitrate, but minimal amounts of ammonia should be

converted because the feed stream to the UV Oxidation unit has a $\text{pH} \leq 7$. Since the oxidation of the organics by peroxide in the UV chambers will produce carbon dioxide, which will cause variability in the pH, the extent of reaction with ammonia will be variable, with more reaction at higher pH (e.g., 8-11) and little to none under the neutral or acidic conditions typical of this point in the ETF flowsheet.

Therefore, no iodine species or ammonium ions are expected to be removed by UV Oxidation (DF = 1).

2.3 Peroxide Decomposer

After peroxide/UV treatment, the stream passes through a bed of granular carbon for destruction of excess peroxide. In the peroxide destruction bed, the iodate would be expected to partially adsorb based on selected literature sources;¹² however, some literature indicates that iodate is not adsorbed by carbon.¹³ The activated carbon bed has been replaced with a manganese dioxide (MnO_2) bed recently, but this unit has not been operated yet. Although it is expected that any iodine would be present as iodate, iodide is known to react with the birnessite mineral form of manganese dioxide ($\delta\text{-MnO}_2$) to form iodate, which will adsorb onto the manganese dioxide under mildly acidic conditions, but adsorption is negligible at neutral pH.¹⁴ It is not known if the manganese dioxide media tested for use in the bed that will be installed in the ETF is the birnessite reactive form,¹⁵ but presumably the media would be birnessite since it is intended to be an oxidative reactant that destroys the excess hydrogen peroxide. Both iodine and iodate are reported to adsorb onto the birnessite, with a capacity of $12.7 \mu\text{mol/g}$ at pH 5.7,¹⁴ and adsorption increases at more acidic pH ranges. Similar observations of oxidation of iodide to iodate and adsorption onto $\delta\text{-MnO}_2$ have also been reported,¹⁶ as well as iodine adsorption.¹⁷ Although the literature indicates that the iodine may be adsorbed onto the new manganese dioxide bed under mildly acidic conditions, it is a strong function of pH, with negligible adsorption at neutral pH and above.¹⁴ The mildly acidic pH of this stream would be expected to cause absorption of iodine onto the bed. However, it is not clear what the effect of the residual hydrogen peroxide in the stream in ETF will have. It is possible that the iodine will be mostly adsorbed onto the manganese dioxide bed, but it may also pass through and exit as iodate ion. The impact of the peroxide decomposer bed on the iodine content of the ETF process stream cannot be quantified at this time and is therefore unknown. Since no upstream ETF unit operations remove iodine species, this process is exposed to the maximum feed iodine concentration, so any tendency to remove iodine would likely result in some accumulation in the media. If it absorbs, over time, the bed will load to saturation and then no removal will occur. To be conservative, a DF of 1 is appropriate but the iodine DF is assigned a value of unknown for the decomposer. However, for future disposition of the peroxide decomposer, it should be noted that it is likely to contain some inventory of ^{129}I . The capacity of this particular form of MnO_2 for iodine absorption under these specific conditions is not known. Although specially prepared MnO_2 has been investigated for use as a catalyst for oxidative destruction of ammonium ion in groundwater applications,¹⁸ these are typically 1-2 mg/L concentrations. The much higher concentration of ammonia in this stream would likely be only negligibly effected by this catalytic reaction. If any reaction occurs, it would form nitrate and/or nitrite, depending on conditions, which are compatible with downstream processing. The peroxide decomposer bed most likely would not have any measurable impact on the ammonium ion content (DF = 1).

2.4 Degas Column (Degasser)

Peroxide destruction is followed by fine particle filtration, and air stripping in a degasification column to remove the carbon dioxide resulting from UV Oxidation (RPP-CALC-64361). The degasification column is packed with polypropylene packing material and operates with counter-current air flow. The iodate would not change speciation or form in the filter or air stripper (see discussion in Section 1.1). The ammonium ion is stable in air and will not change form or partition significantly to the gas phase. As the waste water stream passes through filtration to remove any precipitated solids or carbon fines, it is not expected that any changes occur in the speciation of the iodate. The air from the air stripper passes to the process vessel ventilation system, but it is not expected that any significant iodine or ammonia will be

present in the gas phase at this stage. After these treatment steps, the iodine is expected to remain predominantly as dissolved iodate ions and the ammonia will be present as dissolved ammonium ions.

Therefore, no iodine species or ammonium ions are expected to be removed by the Degas Column (DF = 1).

2.5 Reverse Osmosis

After organics destruction and air stripping, the waste water then passes through a two-stage RO unit (RPP-CALC-64361) with two sets of RO membranes in series. The membranes are Filmtec brackish water membranes. The reject (concentrate) stream volume is 15-25% of the influent amount. Washington River Protection Solutions (WRPS) provided a technical specification for the DOW Filmtec FT-30 membranes used in the facility, which lists a removal efficiency of 97% for sodium iodide (Appendix A). The Filmtec product line ownership has been transferred from Dow to Dupont. Dupont BW30-365 elements are currently in use in the ETF. No information was provided for sodium iodate, but a similar rejection rate to sodium iodide is reasonable to assume.

Rejection rates for ammonium species are not provided on the specification sheet, but pilot plant data is available.¹⁹ The pilot plant data indicates a reject rate of 81.5% for ammonium across the RO system.

Rejection rates of 81.5 and 97% for ammonium and iodide, respectively, correspond to decontamination factors of 5.4 and 33 in the RO system. Therefore, it is expected that the bulk of the iodine and ammonium species would be removed in the Reverse Osmosis unit operation and would be captured in the reject solution that is transferred to the evaporator.

2.6 Ion Exchange

Polishing ion exchange (IX) is performed for the evaporator condensate stream with mixed bed ion exchange columns containing Lewatit MonoPlus S 200 KR cation resin and Lewatit MonoPlus M 800 Anion resin. This polishing step reduces conductivity to less than 0.1 $\mu\text{Si}/\text{cm}$. For the trace amounts of iodine species and ammonium ion that pass the RO membrane, it is expected that the DFs for ammonium and iodine are >100 on these beds. Ammonium ion would be removed by the cation resin and anionic iodine species would be removed by the anion resin. Since the bulk iodine species and ammonium ion removal occurs upstream of the ion exchange beds, this unit operation serves as a polishing step for these species. These small amounts would be removed from the resin during elution and sent to the evaporator in the eluate. This means that this minor portion of the iodine and ammonium species would be captured in the evaporator concentrate and would also flywheel in the system.

2.7 Evaporation of Reverse Osmosis and Ion Exchange Reject

Evaporation of iodine in aqueous solution at neutral or slightly acidic conditions resulted in $\sim 20\%$ of the iodine partitioning to the condensate during VSL evaporations of the melter offgas condensate²⁰ at this approximate pH and the test report states that iodate was the likely form of iodine entering the evaporator. It is likely that some iodine (I_2) was formed during the evaporation process and the iodine partitions to the evaporator condensate. The iodine would be expected to react to form iodide or iodate when blended with the incoming effluent from WTP.

Ammonium is expected to partition predominantly to the concentrated liquor, but a small fraction ($<5\%$) would be expected to partition to the condensate as dissolved ammonia and returned to the front end of the ETF process.

Based on the information in this section and Section 2.5, the bulk of the iodine and ammonium species will be captured in the evaporator concentrate but with some transfer (particularly for iodine) to the condensate.

The iodine and ammonium DFs for the bulk of the stream in the evaporator condensate are estimated to be >20 and ~5, respectively.

2.8 Vessel Off-Gas System

One component in the ventilation system is the High Efficiency Gas Absorber (a.k.a. carbon bed), but this will be removed during a system modification in the near future. Off-gas generated from the ETF will pass through a high-efficiency particulate air (HEPA) filter prior to exiting the facility. The HEPA filter will remove > 99.97 % of airborne particulates from the stream, but little-to-no gaseous species are expected to be removed in this system.

Therefore, no iodine or ammonium species are expected to be removed by the off-gas system (DF = 1).

3.0 Conclusions

The fates of ammonium and iodine in various ETF unit operations has been evaluated based on a review of available data and literature. Estimated decontamination factors for ammonium and iodine for ETF unit operations are shown in Table 3-1. With the exception of iodine in the Peroxide Decomposer, reasonable estimations of the decontamination factors for all operations are provided. Assuming that bulk iodine is not removed in the peroxide decomposer (unknown iodine DF), the bulk of the iodine and ammonium species are expected to be removed from the ETF waste stream in the reverse osmosis reject solution which is concentrated in the evaporator and then cycled back through the surge tank with the incoming ETF feed. Subsequent ion exchange treatment of the ETF process stream is expected to remove the remaining minor amounts of iodine and ammonium species which are transferred in the eluate from the columns to the evaporator. As a result, these minor amounts of iodine and ammonium species are expected to flywheel and accumulate in the ETF process.

Table 3-1. Estimated Decontamination Factors of ETF Unit Operations.

Unit Operation	Ammonium DF	Iodine DF
Filtration	1	1
UV Oxidation	1	1
Peroxide Decomposer	1	Unknown*
Degas Column	1	1
Reverse Osmosis	5.4	33
Evaporation	> 20	≈ 5
Ion Exchange	> 100	> 100
Off-Gas System	1	1

* Depending on the pH, some iodine removal may occur in this unit operation.

4.0 Recommendations, Path Forward or Future Work

Evaluations of iodine removal by the peroxide decomposer system are needed to determine the amount of absorption by that unit.

5.0 References

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Appendix A. DOW FILMTEC Tech Fact Sheet

Tech Fact



FILMTEC™ Membranes

Estimated Percent Rejection of Various Solutes by FILMTEC Membranes

In order to assist customers in estimating the rejection of FILMTEC™ FT30 membranes, tests have been performed with a variety of solute compounds. The results of these tests are indicated as a % rejection for each compound listed in the tables below.

Actual system performance may vary from the listed data, particularly with changes in feed water concentration, pH and temperature. For this reason, these tables should be used as a quick screen. Pilot trials should be performed to determine actual rejection in a specific application.

Solute	MW	Rejection, %
1, 1, 1-Trichloroethane	133	98
1, 2-Dibromoethane	173	15
1, 2-Dichloroethane	99	37
1, 2, 3-Trichlorobenzene	181	>57
1, 2, 4-Trichlorobenzene	181	96
1, 2, 4-Trimethylbenzene	120	57
1, 2-Dichlorobenzene	147	70-92
1, 3-Dichlorobenzene	147	66-69
1, 4-Dichlorobenzene	147	61
1-Chlorododecane	204	87
1-Methylnaphthalene	142	67
2, 2', 5, 5'-Tetrachlorobiphenyl	290	46
2, 4, 6-Trichlorophenol	197	100
2, 4-Dichlorophenol	163	93
2, 6-Dimethylphenol	122	92
2, 6-Di-Tert-Butyl-4-Methylphenol	220	96
3, 8-Dimethylphenol	122	92
3-Hydroxy-Capric Acid	188	>98
3-Pentanone	86	74
4-Ethylphenol	122	84
4-Isopropylphenol	136	84
5-Chlorouracil	146	88
Acetic Acid	60	45
Acetone	58	70
Aluminum Nitrate	213	86
Aluminum Sulfate	342	89
Aniline	93	64-75
Anthraquinone	208	93
Benzene	78	19
Benzoic Acid	122	92
Benzothiazole	133	79
Biphenyl	154	91
Bis (2-Ethylhexyl) Phthalate	390	94
Bromodichloromethane	163	79
Bromoform	94	>67
Cadmium Sulfate	208	97
Caffeine	174	99
Calcium chloride	111	99

Solute	MW	Rejection, %
Calcium Nitrate	164	95
Carbon Tetrachloride	153	98
Cesium Chloride	168	97
Chlorobenzene	112	0-50
Chloroform	119	71-90
cis-1, 2-Dichloroethylene	97	20
Clofibric Acid	214	>99
Copper Sulfate	160	99
Cyclohexanone	98	95
Dibromochloromethane	208	79
e-Caprolactum	113	85
Ethanol	46	38-70
Ethyl Benzene	106	71
Formaldehyde	30	35
Furfural	96	35
Glucose	180	98-99
Glycine	188	78
Heptaldehyde	114	100
Humic Acid		98
Hydrochloric Acid	36	28
Isophorone	138	96
Isopropanol	60	90
Lactic Acid (pH 2)	90	94
Lactic Acid (pH 5)	90	99
Magnesium Chloride	120	98
Magnesium Sulfate	120	99
Manganese (II) Sulfate	151	97
Methanol	32	25
Methyl Ethyl Ketone	72	73
Methyl Isobutyl Ketone	100	98
Naphthalene	128	80
Nickel Chloride	130	96-99
Nickel Sulfate	155	97-99
o-Cresol	108	84
o-Xylene	106	67
p & m Xylene	106	38
Pentachlorophenol	266	>86
Phenol-80%	94	65

Solute	MW	Rejection, %
Phosphoric Acid	96	94
Quinoline	129	97
Silica	60	98
Sodium Acetate (1%)	82	88
Sodium Bicarbonate	84	98
Sodium Bromide	103	96
Sodium Chloride	58	99
Sodium Cyanide	49	95
Sodium Di-H Phosphate	120	98
Sodium Fluoride	42	98
Sodium Hydrogen Sulfate	120	76
Sodium Iodide	150	97
Sodium Mono-H Phosphate	142	98
Sodium Nitrate	85	93-98

Solute	MW	Rejection, %
Sodium Orthophosphate	164	99
Stearic Acid	204	71
Strontium Chloride	158	96
Succinic Acid	118	35
Sucrose	342	99
Sulfuric Acid	98	84
Tetrachloroethylene	165	68-80
Tin (II) Sulfate	215	85
Tributyl Phosphate	266	49
Trichloroethylene	131	30-43
Trimesic Acid	210	96
Urea	60	70
Zinc Chloride	136	93
Zinc Sulfate	161	98

FILMTEC™ Membranes
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Notice: The use of this product in and of itself does not necessarily guarantee the removal of cysts and pathogens from water. Effective cyst and pathogen reduction is dependent on the complete system design and on the operation and maintenance of the system.

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