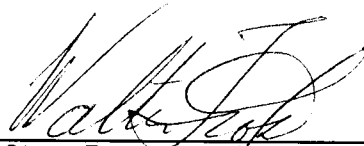


To the Graduate Council:

I am submitting herewith a thesis written by Karen M. Seiser entitled "Identification and Preliminary Design of Dyes and Extraction Techniques for Diffusion Modeling in a Prototype Freon Atmospheric Boundary Layer Simulation Tunnel." I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Engineering Science and Mechanics.



Walter Frost, Major Professor

We have read this thesis and
recommend its acceptance:



Accepted for the Council:



Vice Provost and
Dean of the Graduate School

IDENTIFICATION AND PRELIMINARY DESIGN OF DYES AND EXTRACTION
TECHNIQUES FOR DIFFUSION MODELING IN A PROTOTYPE FREON
ATMOSPHERIC BOUNDARY LAYER SIMULATION TUNNEL

A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville

Karen M. Seiser

December 1986

DEDICATION

This thesis is dedicated to
Mr. and Mrs. William R. Seiser
for their enduring support and understanding.

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ABSTRACT

Diffusion modeling in a water tunnel is a frequently used technique to predict the path and relative concentration of a spilled contaminant in the atmosphere. This technique involves injection of a dye into the tunnel and measurement of its position and concentration along the dispersion path. One illustration of this technique is the prediction of the dispersion path of the space shuttle's plume at Kennedy Space Center, Florida, and Vandenburg Air Force Base, California. Due to the temperature constraints imposed on diffusion modeling in a water tunnel, development of a prototype Freon tunnel is being considered. The types of dye best suited for diffusion modeling in Freon are discussed in this thesis. Combinations of dye and Freon to simulate lighter-than- and heavier-than-air spills are also briefly described. Due to the relative cost of the Freon, various techniques are discussed on how the dye can be extracted such that the Freon is reusable.

Criteria were first established to define those characteristics of dyes best suited for diffusion modeling in a Freon tunnel. Hundreds of dyes were researched for suitability but only those dyes with characteristics meeting the established criteria were recommended for use. Guidelines and illustrations were identified for mixing dyes with various Freons to simulate the range of atmospheric diffusion processes which are of interest for simulation. Several separation and filtration techniques were researched to identify the best and most practical procedure for extracting the dye from the Freon.

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NOMENCLATURE

A	Angstrom micron
C	Degree Celsius
CI	Color index
e	Water vapor pressure
g	Grams
g/l	Grams per liter
g/ml	Grams per milliliter
g/mole	Grams per molecule
gmd	Gallons per meter ² per day
K	Degree Kelvin
ml	Milliliters
mg/l	Milligrams per liter
mm	Millimeter
msec ⁻²	Meters per second ²
P	Pounds per inch
pH	Hydrogen ion activity of a system

Greek Symbols

ρ	Density
--------	---------

CHAPTER I

INTRODUCTION

Diffusion modeling in a water tunnel is a frequently used technique for statistically predicting the behavior of turbulent flows. This technique involves injecting a dye into the tunnel and measuring the position and concentration of the dye as it disperses through the tunnel. One limitation imposed on water tunnels is the inherent difficulty in controlling the temperature of the solution. To alleviate this problem, a Freon tunnel system could be designed. With the advent of a Freon tunnel system on the horizon, several questions have arisen. Some of these questions have been researched and the findings are presented in the following chapters.

In Chapter II the identification of dyes which were found to be best suited for diffusion modeling in a Freon tunnel is discussed. Criteria such as the dye's solubility in the Freon or its light fastness properties were established and used for dye selection. Hundreds of dyes were researched but only those dyes which met the established criteria were recommended for use.

Chapter III provides guidelines and illustrations for mixing dyes with various Freons to simulate lighter-than- and heavier-than-air spills in a Freon tunnel.

Chapter IV addresses various separation and filtration techniques to extract the dye from the Freon after it has been used in the tunnel.

Chapter V summarizes the conclusions and recommendations evolving from this research.

CHAPTER II

DYE IDENTIFICATION FOR DIFFUSION

MODELING IN FREON

A dye is a colored substance that can be applied in solution or dispersion to a substrate thus giving it a colored appearance. The color of a dye is directly related to its visible absorption spectrum, which is in turn closely related to its molecular structure, degree of aggregation, and environment. To identify those dyes best suited for diffusion modeling in a Freon tunnel, the following criteria were established:

1. The dye must be soluble in the substrate;
2. The dye must possess good light fastness properties;
3. The dye must be of the appropriate hue;
4. The dye must not exhibit any tailing effects;
5. The dye must be resistant to temperature excursions; and
6. The dye must be readily/commercially available.

A dye to be selected must be soluble in the Freon such that it realistically simulates the dispersion of hazardous contaminants in the atmosphere. The two general classes of dyes which meet this requirement are: solvent-soluble dyes and disperse dyes. Disperse dyes are a class of substantially water-insoluble dyes which should act as a near-colloidal dispersion in the Freon. Solvent-soluble dyes are a class of organic dyes which are insoluble in water and readily soluble in an organic solvent due to their small size.

Determining whether a mixture is a true solution or a colloidal dispersion is often possible by the method described in Figure 2.1. When light is passed through a true solution, an observer viewing from a direction perpendicular to the beam sees no light, but in a colloidal suspension, light is scattered in many directions and can easily be seen. This behavior, first studied by Tyndall in 1869, is known as the Tyndall effect [1]. Thus, with the utilization of disperse dyes it would be possible to pass a beam of light through the tunnel and observe the diffusion of individual particles.

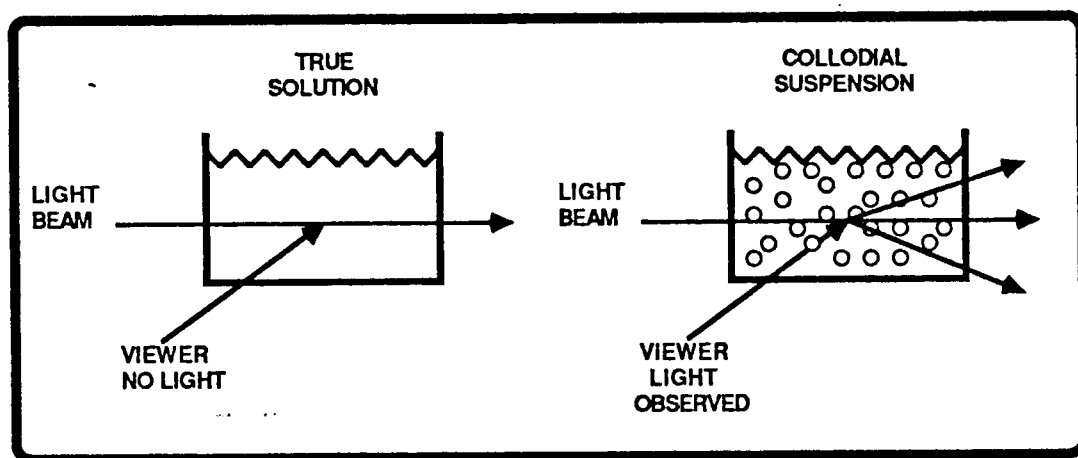


Figure 2.1. Tyndall effect [1].

Solvent-soluble dyes were developed such that they would dissolve in organic mediums. The extent to which the dye will dissolve in Freon is unknown. Therefore, a variety of solvent dyes have been identified for experimentation in the Freon tunnel. It is foreseen that the solubility of each dye in the Freon will vary. Thus, qualitative descriptions describing the various degrees of solubility can be cataloged and serve as an aid in dye selection. Figure 2.2 is

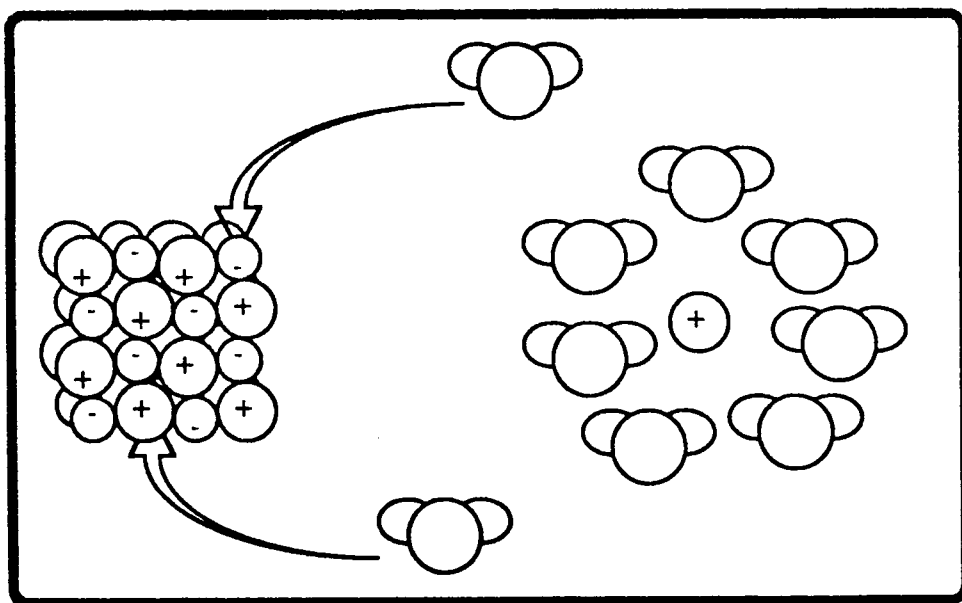


Figure 2.2. Dissolving ionic substances [1].

an illustration of the basic principle of dissolving ionic substances in organic mediums.

To accurately measure the concentration of the dye along the dispersion path, it must possess good light fastness properties; that is, it must resist decolorization due to the influence of light. All of the dyes recommended for use have a light fastness grading of 4 or better. This is based on a grading scale of 1 to 8. Information regarding light fastness grading is discussed in Appendix A. However, these values are based on water as the solvent not Freon. Experimental values of fastness grades in Freon are not available.

The dye must be of the appropriate shade/color (hue) to be used in the tunnel. It would not be advantageous to suggest a light-colored dye for use in a solution of Freon since it would be difficult to distinguish the dye in the tunnel. Thus, dyes with dark hues that contrast with the Freon such as red, brown, blue, and black are

recommended over lighter, less contrasting hues such as yellows and oranges.

Another problem in using dye is the tailing effect. Tailing is defined as the gradual and progressive change in color of the dye as time progresses. A change in color of the dye in the Freon would make it difficult to measure the concentration of the dye along the dispersion path. Thus, dyes which are known to exhibit tailing effects are not recommended.

The purpose of the Freon tunnel is to model atmospheric dispersions within the temperature limits of the Freon. Given in Table 2.1 is a partial listing of some Freons for use in the tunnel and their physical properties [3]. Thus, all of the dyes recommended are compatible within the temperature ranges of the Freons. Compatibility is based on the fact that the dyes are manufactured at temperatures

TABLE 2.1. Physical Properties of Various Freons [3].

Common Name	Formula	Formula Weight (g/mole)	Density (g/ml)	Melting Point C	Boiling Point C
Freon 113	1,1,2,Trichloro-1,2,2,Trifluoroethane	187.38	1.564	-36.4	47.6
Freon 22	Chloro-difluoro methane	86.47	1.510	-146.0	-40.8
Freon 13	Chloro-trifluoro methane	104.46	1.470	-181.0	-81.1
Freon 12	Dichloro-difluoro methane	120.91	1.750	-158.0	-29.8
Freon 21	Dichloro-fluoro methane	102.92	1.405	-135.0	9.0

which exceed the boiling point of Freons and that they should not decompose at various temperatures.

Finally, the dyes must be readily/commercially available. This criteria did not play a substantial role in dye selection since all of the dyes suggested for use could be obtained through one of the following dyestuff companies:

- Crompton & Knowles Corporation
Industrial Products Division
500 Pear Street
P.O. Box 341
Reading, PA 19603
(312) 675-5510
- BASF Wyndotte
Pigment Dept.
491 Columbia Avenue
Holland, MI 49423
(616) 392-2391
- Ciba-Giegy Corporation
Dyestuff & Chemicals Division
Greensboro, NC 27491
(800) 334-5580
- Atlantic Chemicals
Kingsland Station Street
Nutley, NJ 07110
(800) 631-7282

No information could be found on the behavior of dyes in Freon. Thus, listed in the following tables are dyes which are known to meet the established criteria in aqueous and, in some cases, organic solutions.

The tables provide basic information necessary to order a dye through a dyestuff company, such as the color index (CI) reference number, the common name of the dye, the chemical class which the dye belongs to, and its hue. In Appendix B, information regarding the

preparation of the dyes, their structure, and some physical properties are given.

1. CLASS 1: SOLVENT-SOLUBLE DYES

Solvent dyes are derived mainly from the various cationic classes. This class of dyes is distinguished by the presence of basic groups in their molecules and, thus, have an affinity for protein fibers and cellulosic fibers [2]. The azo and anthraquinone classes are also represented. In general, the azo class supplies yellow, orange, and red hues; the cationic dye supplies orange to green hues; and the anthraquinone class supplies violet to green hues.

Cationic dyes are usually free bases which correspond in the dye of the basic application class. Table 2.2 lists the various chemical classes of cationic dyes, their CI reference number, the common name of the dye class, and the hue. Various chemical classes are represented, all characterized by exceptional brilliance and good fastness to light. Only those dyes which meet the established criteria outlined in this chapter are listed.

The use of fluorescent dyes in the Freon tunnel has also been explored. Of the dyes listed in Table 2.2, fluorescent dyes come from the xanthene class. One of the earliest xanthene dyes was fluorescein (CI acid yellow 73: CI45350). Its strong green fluorescence in solution, even at great dilutions, enables fluorescein to be used as a marker for life saving at sea, for tracing the course of underground streams, and in medical practice for detection of weak circulation of blood. It has been discovered that m-dialkyl aminophenols can be

TABLE 2.2. Solvent-Soluble Dyes [2].

Chemical Class	CI Reference Number	Common Name	Hue
Diphenylmethane	CI solvent yellow 34 (CI 41000B)	Auramine O	Bright greenish-yellow
Acridine	CI solvent orange 15 (CI 46005B)	Acridine orange	Bright orange
Xanthene	CI solvent red 49 (CI 45170B)	Rhodamine B	Bright bluish-red
Triarylmethane	DI solvent red 41 (CI 42510B)	Magenta	Bright bluish-red
Triarylmethane	CI solvent violet 8 (CI 42535B)	Methyl violet	Bright bluish-violet
Triarylmethane	CI solvent violet 9 (CI 42555B)	Crystal violet	Bright bluish-violet

TABLE 2.2. (Continued),

Chemical Class	CI Reference Number	Common Name	Hue
Triarylmethane	CI solvent blue 3 (CI 42775)	Spirit blue	Blue
Triarylmethane	CI solvent blue 4 (CI 44045B)	Victoria blue B	Bright blue
Indophenol	CI solvent blue 22 (CI 49705)	Fat blue Z	Blue
Induline	CI solvent blue 7 (CI 50400)	Induline	Reddish-blue
Nigrosine	CI solvent black 5 (CI 50415)	Nigrosine spirit soluble	Bluish-black
Nigrosine	CI solvent black 7 (CI 50415B)	Nigrosine base	Bluish-black

condensed with phthalic anhydride to give dyes that surpass that of fluorescein in their light fastness; these dyes are the rhodamines. Their shades are strong, bright bluish-reds with a strong fluorescence. Sulphonated derivatives of the rhodamines provide acid dyes with good fastness to light. An example is coomassie violet 2R (CI acid violet 9: CI 45190). This dye gives bright reddish shades with good light fastness properties [2].

Table 2.3 lists examples of some azo-solvent dyes which have met the established criteria. Most of the azo-solvent dyes contain only one azo group and thus provide yellow and orange hues, but diazo dyes provide a few red and brown shades.

Several commercial solvent dyes consist of salts formed between a cationic dye and an acid dye; the latter usually being in the form of a chromium complex. Products of this generally have good fastness to light and thus show a great advantage over cationic dyes used alone. CI solvent red 35 consists of the salt of rhodamine B (CI 45170) with the chromium complex of 2-amino-4-chlorophenol-2-naphthol-6; 8-disulphonic acid (CI 16260) is an example. It gives bright bluish-red shades with good fastness to light [2].

Solvent dyes of the anthraquinone series include various N-alkyl, N-aryl, N-alkyl-N'-aryl, N, N'-dialkyl, and N, N'-diaryl derivatives of 1,4-diamino anthraquinone. An example is CI solvent green 3 (CI 61565) which is 1,4-di-p-toluidino-anthraquinone. This dye gives bluish-green shades with good fastness to light. If the anthraquinone is substituted in the one and four positions by an N-arylamino group, a dye is obtained with greatly increased solubility in a hydrocarbon

TABLE 2.3. Azo-Solvent Dyes [2].

CI Reference Number	Hue
CI solvent yellow 1 (CI 11000)	Reddish-yellow
CI solvent yellow 2 (CI 11020)	Reddish-yellow
CI solvent yellow 14 (CI 12055)	Bright reddish-yellow
CI solvent orange 1, CI food orange 3 (CI 11920)	Yellowish-orange
CI solvent orange 2 (CI 12100)	Reddish-orange
CI solvent orange 3 (CI 11270B)	Dull yellowish-orange
CI solvent orange 7 (CI 12140)	Reddish-orange
CI solvent red 4 (CI 12170)	Yellowish-red
CI solvent red 23 (CI 26100)	Yellowish-red
CI solvent red 24 (CI 26105)	Red
CI solvent brown 1 (CI 11285)	Reddish-orange
CI solvent brown 5 (CI 12020)	Dull brown
CI solvent brown 12 (CI 21010B)	Dull yellowish-red
CI solvent red 8 (CI 12715)	Bright bluish-red
CI solvent violet 1 (CI 12196)	Dull bluish-violet

solvent. An example is calco gas blue NA (CI solvent blue 14: CI 61555) [1]. Other dyes of this nature are listed in Table 2.4.

TABLE 2.4. Derivatives of the 1,4-Diaminoanthraquinone Class [4].

Dyes in Which the 1,4 Positions are Filled by Arylamino Groups	
Common Name	Hue
Alizarine cyanine green G (CI 1078)	Bluish-green
Alizarine viridine FF	Bluish-green
Alizarine cyanine green 5G	Yellow-green
Alizarine supra blue SE	Bright blue
Alizarine brilliant green G	Clear green

2. CLASS 2: DISPERSE DYES

Disperse dyes that could be used in a Freon tunnel come from two classes: (1) azo-disperse dyes and (2) anthraquinone-disperse dyes. Examples of azo-disperse dyes which meet the established criteria are given in Table 2.5. Violet and blue hues are included, but disperse dyes of these shades are derived mainly from the anthraquinone series. In 1923 the British Dyestuff Corporation developed the azo-disperse dyes. This firm also produced a series of anthraquinone-disperse dyes, and these are sold under the name Duranol. Many other firms have manufactured dyes of the same type, mostly fairly simple 1-amino-anthraquinone derivatives carrying substituents such as alkyl, aryl,

TABLE 2.5. Azo-Disperse Dyes [2].

CI disperse yellow 3 (CI 11855)
CI disperse yellow 4 (CI 12770)
CI disperse orange 1 (CI 11080)
CI disperse orange 3 (CI 11005)
CI disperse orange 5 (CI 11100)
CI disperse orange 13 (CI 26080)
CI disperse red 1 (CI 11110)
CI disperse red 13 (CI 11115)
CI disperse violet 7 (CI 11410)
CI disperse blue 38 (CI 11430)
CI disperse black 1 (CI 11365)

TABLE 2.6. Anthraquinone-Disperse Dyes [2].

CI Reference Number	Typical Dye	Hue
CI disperse orange 11 (CI 60700)	Duranol orange G	Bright orange
CI disperse red 11 (CI 62015)	Duranol red X3B	Bright bluish-pink
CI disperse red 15 (CI 60710)	Duranol red 2B	Bluish pink or red
CI disperse violet 1 (CI 61100)	Duranol red 2R	Bright violet
CI disperse violet 4 (CI 61105)	Celliton fast violet 6B	Bright bluish violet
CI disperse blue 1, CI solvent blue 18 (CI 64500)	Duranol brilliant blue	Blue
CI disperse blue 3 (CI 61505)	Duranol brilliant blue	Bright blue
CI disperse blue 6 (CI 62050)	Celliton fast blue FFG	Bright blue

aralkyl, or hydroxyalkyl groups. Table 2.6 lists anthraquinone-disperse dyes suitable for use in the Freon tunnel. The light fastness of anthraquinone-disperse dyes is good with an standard dye concentration (SDC) rating mainly in the region 5 to 6, rather better than the average given by the azo-disperse dyes [4].

CHAPTER III

GUIDELINES ON SPILL SIMULATION

To simulate lighter-than- and heavier-than-air spills, it is essential that the injected dispersion solution in the Freon tunnel be less dense than the Freon in the tunnel in the case of lighter-than-air spills and more dense in the case of heavier-than-air spills. The injected solution will be a mixture of various quantities of dye and Freon to achieve the appropriate density. A functional flowchart illustrating the guidelines and the approach utilized in obtaining the desired dye and Freon mixture is presented in Figure 3.1.

A step-by-step breakdown of the procedure follows:

Step 1. Calculate the density of the air in the spill environment (ρ_{air}). This value depicts the relative temperature, pressure, and humidity at the contamination site. The density of the dry air may be determined from the following equation:

$$\rho = \rho_0(T_0/T)(P/P_0)$$

where

$$\rho_0 = 1.2929 \text{ g/l}$$

$$T_0 = 273.3 \text{ K}$$

$$P_0 = 760 \text{ mmHg}$$

T = temperature at the contamination site

P = pressure at the contamination site.

If the air in the spill environment is moist, the density may be determined in a similar fashion:

$$\rho = \rho_0(T_0/T)(P_B - 0.378e)/P_0$$

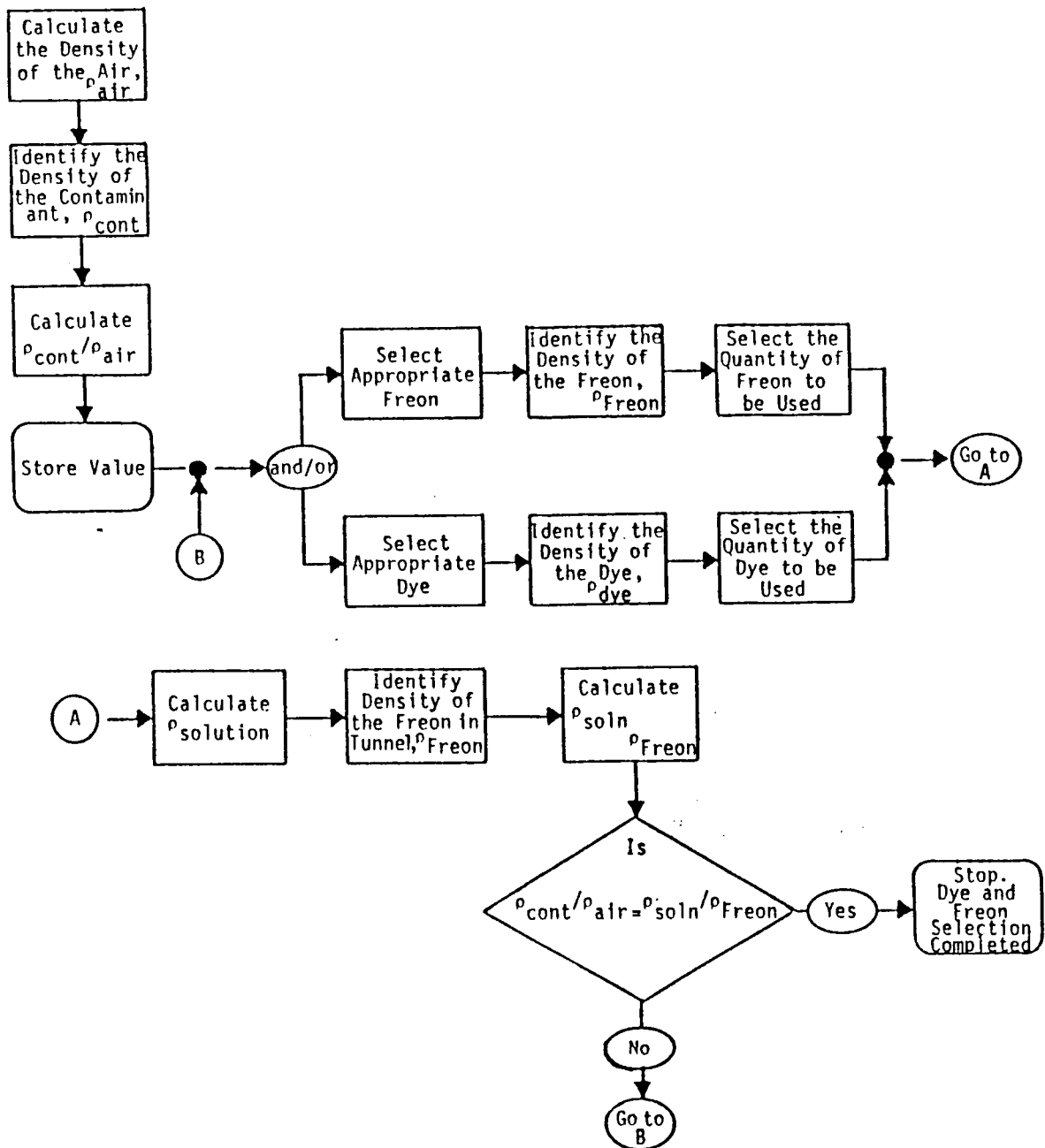


Figure 3.1. Functional flowchart of guidelines for dye and Freon selection.

where

P_B = barometric pressure at the contamination site

e = vapor pressure of the moist air.

Step 2. Identify the density of the spilled contaminant (ρ_{cont}).

Step 3. Calculate ρ_{cont}/ρ_{air} . Store this value.

Step 4. Select a dye and Freon that are appropriate for the requirements of the atmospheric process to be simulated.

Step 5. Identify the density of the dye (ρ_{dye}) and Freon (ρ_{Freon}) selected.

Step 6. Select a quantity of dye (V_{dye}) and Freon (V_{Freon}) to be used as the dispersion solution.

Step 7. Calculate the density of the injection solution (ρ_{soln}).

$$\rho_{soln} = \frac{\text{mass}_{Freon} + \text{mass}_{dye}}{V_{dye} + V_{Freon}} \quad (V_{soln} = V_{dye} + V_{Freon})$$

Step 8. Identify the density of the Freon to be used in the tunnel (ρ_{Freon}). Freon 113 has been initially selected for utilization in the tunnel. It has a density of 1.546 g/ml.

Step 9. Calculate ρ_{soln}/ρ_{Freon} .

Step 10. If $\rho_{soln}/\rho_{Freon} = \rho_{cont}/\rho_{air}$, then a dye and Freon combination have been identified to simulate the desired conditions. If the ratio is not acceptable for the desired accuracy, then return to Step 4 and select a different combination of dye and Freon or return to Step 6 and change the quantity of dye and Freon to be used. Repeat the steps until the desired accuracy has been obtained.

It would be possible to implement a computer program to identify the proper dye and Freon combination. The computer would be able to evaluate various dye and Freon combinations to achieve a more accurate estimate of $\rho_{\text{cont}}/\rho_{\text{air}} = \rho_{\text{soln}}/\rho_{\text{Freon}}$ in a much smaller time frame than the manual approach. Table 2.1, page 5, serves as an aid in Freon selection.

To illustrate the guidelines previously outlined, a dye and Freon combination will be identified for use in the simulation of a lighter-than- and a heavier-than-air spill. The evaluation of the density of the contaminant divided by the density of air, which is approximately the density of the solution divided by the density of the Freon, will not be addressed.

The general requirement of determining the dye and Freon combination (injection solution) that is less dense (lighter than air) or more dense (heavier than air) than the Freon in the tunnel will be illustrated. Since Freon 113 (1,1,2,-trichloroethane, 1,2,2-trifluoroethane) will be the primary solvent utilized in the tunnel, injection solutions of a greater and lesser density than that of Freon 113 will be identified.

The density of the injection solution is determined from the following equation:

$$\rho_{\text{soln}} = \frac{\text{mass}_{\text{dye}} + \text{mass}_{\text{Freon}}}{V_{\text{soln}}} \quad (3.1)$$

The dye to be utilized in both illustrations is CI disperse red 11, an anthraquinone-disperse dye with a density of 1.438 g/ml. In both illustrations, one liter of injection ($V_{\text{soln}} = 1.01$) solution will

be made; however, it should be noted that the guidelines identified are applicable to any size solution.

Lighter-than-air spills are determined from the following equation:

$$\rho_{\text{soln}} < \rho_{\text{Freon}} = 1.564 \text{ g/ml.} \quad (3.2)$$

To obtain a solution density less than 1.564 g/ml, the dye should be mixed with a Freon of lesser density than Freon 113. From Table 2.1 it can be seen that dichlorofluoro-methane meets this requirement with a density of 1.405 g/ml. However, it should be noted that the boiling point of Freon 21 is approximately 39° below the boiling point of Freon 113. Thus, it may not be possible to mix the two Freons unless the temperature is kept below 9° C in the tunnel.

The mass of the dye has been selected to be 0.50 grams, which is the first evaluation of ρ_{soln} . This amount should be sufficient to provide a significant color change. However, experimentation may prove otherwise; and thus, the amount may need to be increased.

$$\text{mass}_{\text{dye}} = 0.50 \text{ g; volume}_{\text{dye}} = 0.35 \text{ ml;}$$

$$\text{volume}_{\text{Freon}} = 100 \text{ ml} - 0.35 \text{ ml} = 99.65 \text{ ml;}$$

$$\text{mass}_{\text{Freon}} = (1.405 \text{ g/ml})(99.65 \text{ ml}) = 140.01 \text{ g.}$$

Thus,

$$\rho_{\text{soln}} = \frac{0.50 \text{ g} + 140.01 \text{ g}}{100 \text{ ml}} = 1.41 \text{ g/ml.} \quad (3.3)$$

To obtain a less dense injection solution, the amount of dye in the mixture is decreased.

Heavier-than-air spills are determined from the following equation:

$$\rho_{\text{soln}} < \rho_{\text{Freon}} = 1.564 \text{ g/ml.} \quad (3.4)$$

To obtain a solution with a density greater than 1.564 g/ml, the dye should be mixed with a Freon of greater density than that of Freon 113. From Table 2.1 it can be seen that dichloro-difluoro-methane (Freon 12) meets this requirement with a density of 1.75 g/ml. Again, however, it should be noted that the boiling point of Freon 12 is 29.8° C, approximately 78° below that of Freon 113. Thus, it may only be possible to mix the two Freons when the temperature is at or below 29.8° C.

First, evaluation of ρ_{soln} is given as:

$$\text{mass}_{\text{dye}} = 0.50 \text{ g; volume}_{\text{dye}} = 0.35 \text{ ml}$$

$$\text{volume}_{\text{Freon}} = (1.75 \text{ g/ml})(99.65 \text{ ml}) = 174.39 \text{ g.}$$

Thus, $\rho_{\text{soln}} = 1.75 \text{ g/ml}$ using Equation 3.3.

Theoretically, it is possible to increase the density of the injection solution by decreasing the amount of dye added. However, it can be seen that the density of the solution is dominated by the Freon, and the dye plays a very minor role in increasing or decreasing the density, only if a small amount of dye is required to color the solution (0.50 g).

To obtain the desired density in the injection solution, any one or combination thereof of the following factors may be altered:

1. Selection of the dye,
2. Selection of the Freon,
3. The quantity of dye and Freon, and
4. The volume of the injection solution.

CHAPTER IV

DYE EXTRACTION TECHNIQUES

With such a large variety of filtration systems and techniques commercially available, this thesis will only address those techniques which are suitable for separating dyes with a molecular diameter of 10^{-3} to 12^{-3} microns (10 to 12 Å) from Freon. The following separation techniques will be discussed:

1. Ultrafiltration by reverse osmosis,
2. Separation by loose powders,
3. Dye retention on fibers, and
4. Freon distribution.

The relative particle size spectrum for various membrane filtration techniques is shown as a function of filtration flux in Figure 4.1. At the small-molecule/low-flux end of the spectrum lies the commercial cellulose acetate reverse osmosis membrane with the capability of retaining hydrated sodium and chloride ions. Next come ultrafiltration membranes with pores that span a size range of about 10^{-3} to 10^{-2} microns (10 to 100 Å) with filtration fluxes of about 0.5 to 10 gallons per 0.3048 square meters per day (gmd) per pound per square inch (psi) of pressure driving force (F). Microporous filters capable of virus and bacteria retention cover the size range of about 0.01 to 1.0 microns (100 to 10,000 Å) with fluxes of 10 to 1000 gmd per psi (P). Finally, conventional industrial filters for normal particulate materials are capable of filtering particles of 1 micron or larger with filtration fluxes about 1000 gmd per psi of pressure loss [5].

Due to the relatively small molecular diameter of the dyes ranging from 10^{-3} to 12^{-3} microns (10 to 12 A), it can be seen from Figure 4.1 that an ultrafiltration technique will be necessary for extraction of the dyes from the Freon. Since the conventional particle filters and microporous filters have no useful applications for the filtering requirements stated herein, these techniques will not be addressed.

1. ULTRAFILTRATION OF REVERSE OSMOSIS

Ultrafiltration is a membrane filtration process which separates "particles" of about 10 to 100 A from their surrounding medium. The basic principle of operation of ultrafiltration is illustrated in Figure 4.2. Flowing over the membrane is a solution containing two solutes: one of molecular size too small to be retained by the membrane and the other of larger size for which 100 percent retention is possible. A hydrostatic pressure is applied to the upstream side of the supported membrane, and solvent plus small-molecule solute pass through the membrane while the large-molecule solute may be theoretically retained (rejected) 100 percent by the membrane. However, most commercial applications operate at less than 100 percent retention to obtain an optimum flow rate. A fluid concentrated in the retained solute is unpermeable to the membrane, and the solution of small-molecule solute is collected from the downstream side as it passes through the membrane.

Ultrafiltration membranes can contain pores of a relatively narrow size distribution. Because of the small pore size in

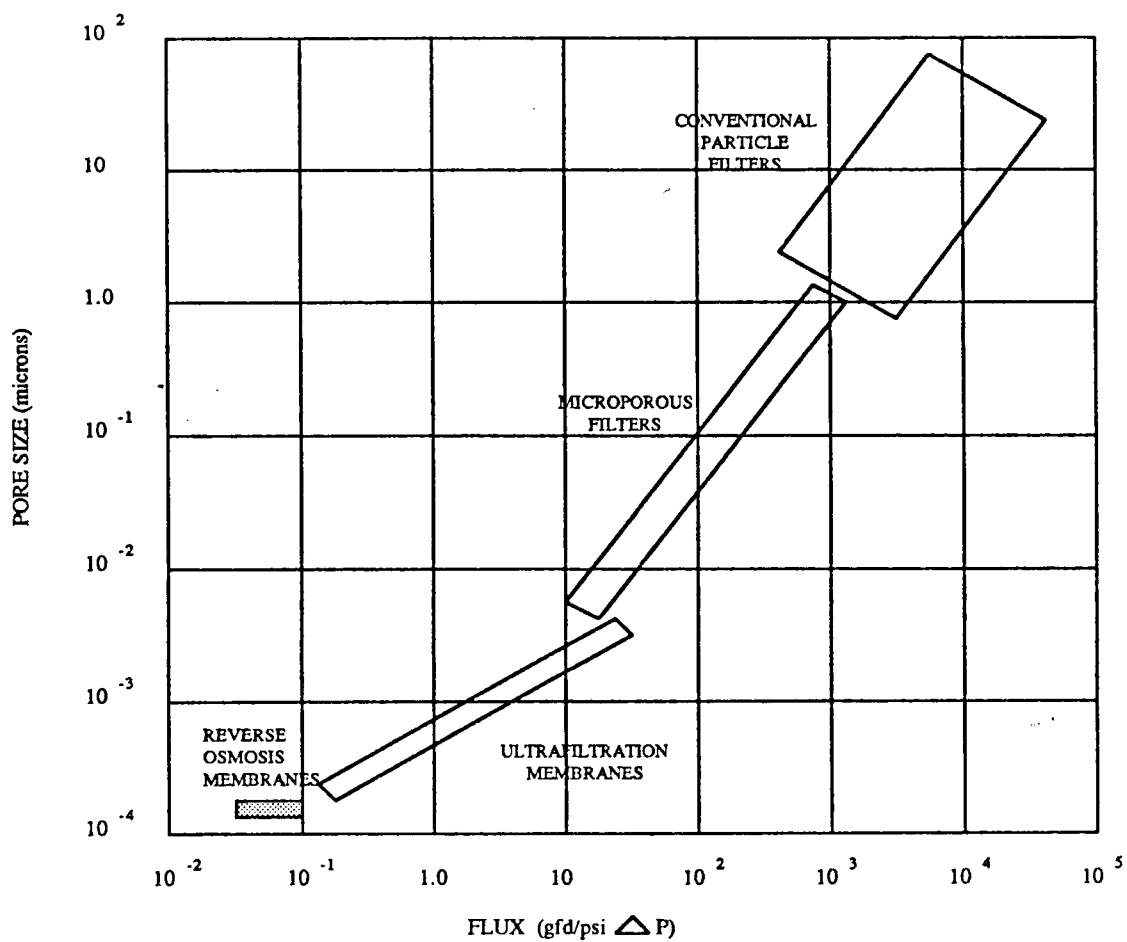


Figure 4.1. Pore size versus flow rate for separation media [5].

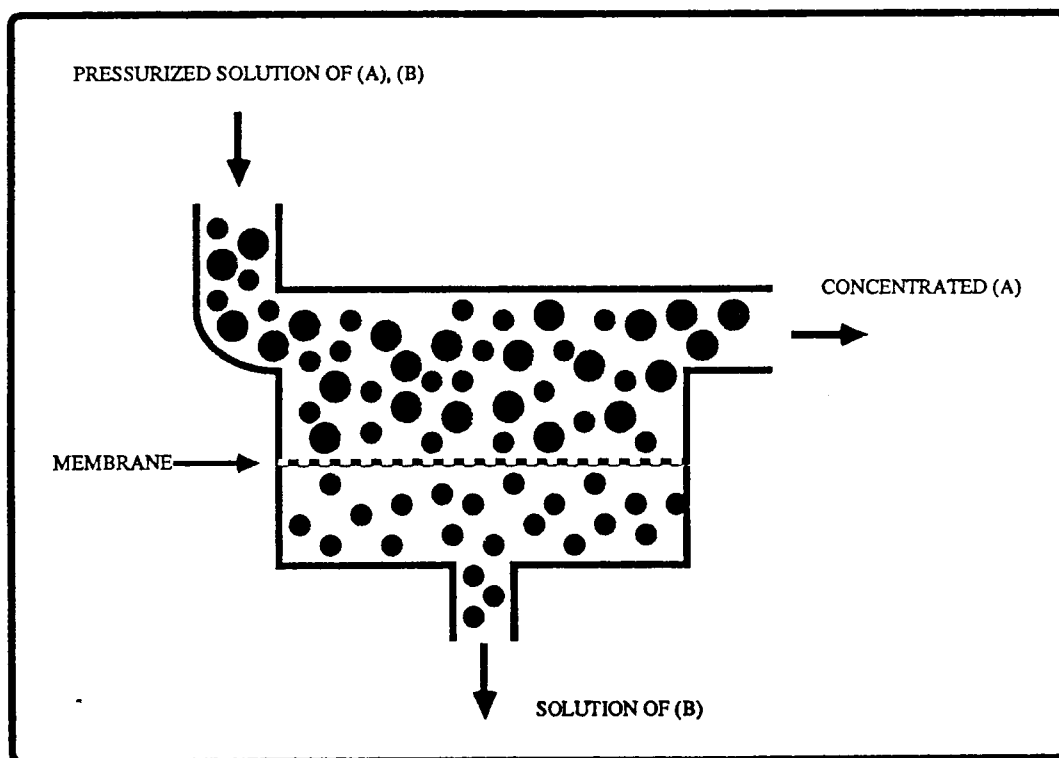


Figure 4.2. The principle of ultrafiltration [5].

filtration membranes, effects of external conditions on the solid membrane material can have an effect on the porous structure. For example, changes in temperature which would change the properties of the membrane due to alterations in swelling properties may cause changes in the selective properties of the membrane. Similarly, in aqueous or Freon systems, extremes in pH may affect the chemical composition of the membrane and thus alter its ultrafiltration characteristics [5].

Relative to other filter media, ultrafiltration membranes are fragile and require supporting substrates, a need which is accentuated by the higher pressure differences used because of low permeability

compared to more porous filter media. Membranes are also sensitive to puncture or mechanical abrasion, which can destroy selectivity [6].

The majority of ultrafiltration membranes are based on synthetic film-forming organic polymers, such as polyamides, polysulphones, polyacrylonitrile copolymers, or cellulose derivatives. The membranes themselves consist of an integral, asymmetric two-layer structure. The side of the membrane in contact with the pressurized feed solution consists of a skin layer, about 0.2 microns in thickness, which contains the fine pores at which separation takes place. The remainder of the membrane thickness consists of a coarsely porous sublayer, which supports the skin, giving a total thickness of about 100 microns. Industrial membranes are also capable of continuous operation over periods ranging from one to three years [5].

In a typical ultrafiltration process, the molecular weight of the solute should be approximately tenfold that of the solvent to obtain desirable retention of the solute.

Therefore, since the dyes to be filtered have a molecular weight in the range of 200 to 1000 g/mole, which is nowhere near 2000 g/mole (tenfold the molecular weight of Freon 113), a reverse osmosis system should be applied. Separation by reverse osmosis is considered to be an application of the ultrafiltration membrane process.

Reverse osmosis has been the only technique, in principle, to effectively separate dyes from the solvent in commercial dye houses. The other possible technique is carbon absorption, but it does not remove dissolved solids. Reverse osmosis will not only remove the dye colors, but will also remove 95 percent of the total dissolved solids [6].

It should be noted that Carre, Inc. (207 North Fairplay Street, Seneca, SC 29678) has initiated a program to demonstrate high-temperature hyperfiltration (reverse osmosis) to renovate hot industrial waste water for direct recycling. They are installing a 10 m/hr membrane system to achieve closed-cycle operation of a textile dye range. Hydrous-zirconium oxide-polyacrylate membranes dynamically formed on porous sintered stainless steel tubes arranged in a single-pass system will be supplied by the Mott-Brandon Corporation. The 82° C (180° F) dye wash water contains residual dyes, auxiliary detergent and wetting agents, and guar gum. The total solids in the wash water range from 130 to 7000 mg/l with the suspended solids ranging from 2 to 90 mg/l. The membrane system will be operated at 96 percent recovery [7].

A proposed reverse osmosis system designed to filter the dye from the Freon is illustrated in Figure 4.3. The Freon would be stored in a reservoir and then pumped into the tunnel where the dyes are injected. When the diffusion modeling has been completed, the dye and Freon are pumped back to the reservoir. From this reservoir tank the contaminated Freon would then be passed over a sand filter to remove any particulates other than the dye in the Freon. The Freon would then pass through a reverse osmosis unit where the dye would be collected, and the pure Freon would be pumped back to the reservoir for further use in the tunnel. This filtration system would represent a continuous flow. The membrane of the reverse osmosis unit would consist of cellulose acetate which is capable of withstanding the temperature range which the Freon tunnel would exhibit. (The estimated cost of a

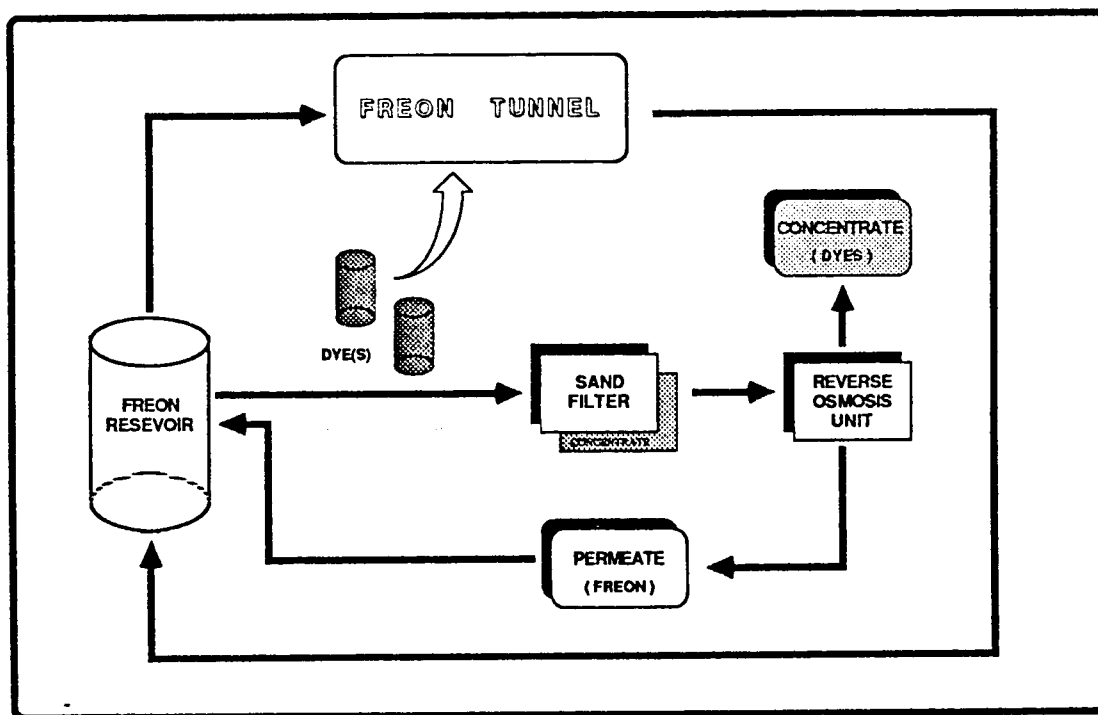


Figure 4.3. Reverse osmosis filtration system.

reverse osmosis unit of this type is on the order of \$250,000, based on personal communications with Carre, Inc., one of the leading manufacturers of reverse osmosis units, based on 1985 dollar value.)

2. LOOSE-POWDER FILTRATION

There is a wide variety of loose powders which are used in liquid filtration in three distinctly different fashions:

1. As thin layers, or precoat, on flexible or rigid media.
This is a convenient way to trap very fine particles which would pass through a rigid or flexible medium.
2. As deep beds, when the purpose may be either to filter in depth, as in a sand filter, or it may be to contact

the liquid with the solid material of the bed so as to enable an absorption process to occur.

3. As additives dispersed in the liquid to be filtered, when the purpose may be either to carry out an absorption process or to increase the porosity of the cake of solids formed on the filter.

There are five major classes of loose-power filter aids commercially available:

1. Diatomaceous Earth,
2. Expanded perlite,
3. Inactive carbon,
4. Absorbent powders, and
5. Coarse materials.

A discussion of each class follows.

Class 1: Diatomaceous Earth. Diatomaceous Earth or diatomite is the classic material to use either as a precoat or body aid. There is some variation in the chemical composition of the various competitive grades of diatomite on the market, but the differences are generally small. The particle size analysis is far more prone to variation and will range from 1 to 100 microns [8]. The Great Lakes Carbon Corporation has performed much work to measure the ability of their different grades of filtration aids to trap particles of a known size ranging from 0.1 to 10 microns. Their results are presented in Figure 4.4.

Class 2: Expanded Perlite. Perlite is a rock of much the same composition as granite and, like granite, is of volcanic origin. The expanded perlite used as a filter aid is prepared from the mineral by a

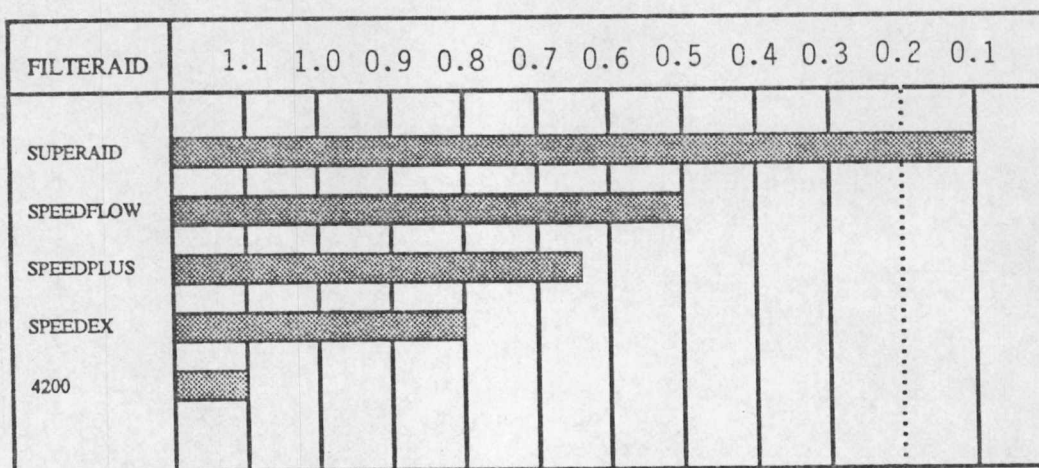


Figure 4.4. Relative efficiency of various filter aids [7].

sequence of operations combining crushing, grinding, and screening. By careful control of the manufacturing process, the density of the expanded perlite can be varied almost at will. The major application of expanded perlite is on rotary vacuum precoat filters. Measured on the basis of filtration rates, there are grades of expanded perlite available corresponding to the major part of the flow range of diatomites. Table 4.1 gives the typical particle size analyses for a range of diatomite and perlite filter aids. From the table it can be seen that the expanded perlite has a much poorer performance in the removal of submicron particles. The relative lack of surface area may account for the poor performance. Another limitation of expanded perlite is that it is not suitable for use over quite so wide a range of diatomite, generally being confined to a zone of pH 4 to 9; diatomites can resist slightly higher levels [8].

Class 3: Inactive Carbon. In the last ten years, several filter aids have been developed from carbon using coal as the ultimate raw material. An example is "Nerofil," which was developed in the

TABLE 4.1. Comparison of Expanded Perlite to Diatomite [7].

Typical Screen Analysis of Clarcel Range of Expanded Perlite Filter Aids								
Grade	0-5u	5-10u	10-20u	20-30u	30-50u	50-100u	100-150u	<150
FLO CR	3	4	8	6	19	34	14	12
FLO 1	4	4	10	8	20	30	16	8
FLO 2	7	9	29	12	17	12	13	1

TABLE 4.1. (Continued)

Typical Particle Size Analysis for Range of Diatomite Filter Aids							
Quality	0-5u	5-10u	10-20u	20-30u	30-50u	50-100u	>100
CB	25	23	24	6	13	8	1
DC	12	20	45	9	2	5	7
DIP	23	47	25	3	2	0	0
DIF	4	28	49	12	4	2	1
DIC	5	14	43	15	10	7	6
DIC-2	5	14	43	15	10	7	6
DIC-S	5	13	43	16	10	7	6
DIC-3	4	9	35	19	13	13	7

United States. Although it is no longer made in the United States, the process has been licensed to a German company who produces an identical material under the name "Synofil." These materials are available in several grades, all of which correspond broadly with the coarse end of the range of diatomites and perlites. The special value of these carbon-based materials is that they can safely handle higher alkaline solutions in which the other silica-based filter aids tend to dissolve [8].

Class 4: Absorbent Powders. This group covers a miscellaneous assortment of materials such as Fuller's Earth, bentonite, activated and unactivated carbons. Strictly speaking, none of these powders are filter mediums or filter aids, but since they are finely divided powders or granular solids, physically they are quite capable of functioning as filter aids. However, if used solely for this purpose, they would generally be found to be decidedly uneconomical. These materials are all absorptive solids; that is to say, they are able to extract certain compounds from liquids or gases by the process of absorption, much as blotting paper soaks up ink. For example, Fuller's Earth is used in this way to remove color and acidity from mineral oils; a certain amount of filtration, whereby fine solid particles are removed, is achieved as the oil flows through the bed of earth [8].

Class 5: Coarse Materials. Many different crushed and granular materials have been used to make up the type of deep beds employed in the large gravity and pressure filters. These materials include crushed quartz, crushed marble, charcoal, coke, anthracite, and silica sand. Experiments over the years have confirmed silica sand to be the

most satisfactory filtration material so that silica sand is the standard filter medium used in modern practice. The sizes of sand and gravel typically used in graded layers in a filter are indicated in Table 4.2. The term sand is generally applied to materials below 0.3175 cm and gravel for larger.

The effective size is intended to indicate the minimum size of the bulk of the sand, and is expressed in millimeters (mm). The size grading of the sand can be chosen to give finer or coarser filtrations as each circumstance demands. Similar deep beds have been used to remove particles ranging from 0.1 to 3 microns with an efficiency of 95 percent or greater in some instances. Beds of carbon granules have also found wide use in sterilizing air for aerobic fermentation processes such as in the manufacturing of penicillin. It has been reported that a 15.24 cm bed of 8 to 14 mesh carbon operating with a superficial air velocity of $0.1524 \text{ m sec}^{-2}$ was so efficient that it was penetrated by only one micro-organism/100,000 [9].

A suggested coarse material bed consisting of activated carbon to filter the dye from the Freon is illustrated in Figure 4.5. The pure Freon is circulated through the tunnel and gradually increases in contamination. When the diffusion modeling has been completed, the contaminated Freon is then filtered. The contaminated Freon is passed over a coarse material bed of activated carbon where the dyes are removed from the Freon. The pure Freon then travels back to the reservoir for further use in the tunnel. (The average cost of activated carbon is on the order of \$1.35 per pound, and the estimated cost of a 12 x 6 x 6 inch (30.48 x 15.24 x 15.24 cm) bed of activated

TABLE 4.2. Typical Range of Sands Used in Deep Bed Filters [7].

Grade	Effective Size (mm)
-16 + 30 (1.0-0.55 mm)	0.55-0.65
-1/8 in + 16 (3.18-1.0 mm)	1.10-1.40
-1/4 in + 1/8 in (6.35-3.18 mm)	3.20-3.60
-1/2 in + 1/4 in (12.7-6.35 mm)	6.40-7.20
-3/4 in + 1/2 in (19.05-12.7 mm)	12.80-13.60
-1 1/2 in + 3/4 in (38.5-19.05 mm)	19.50-21.60

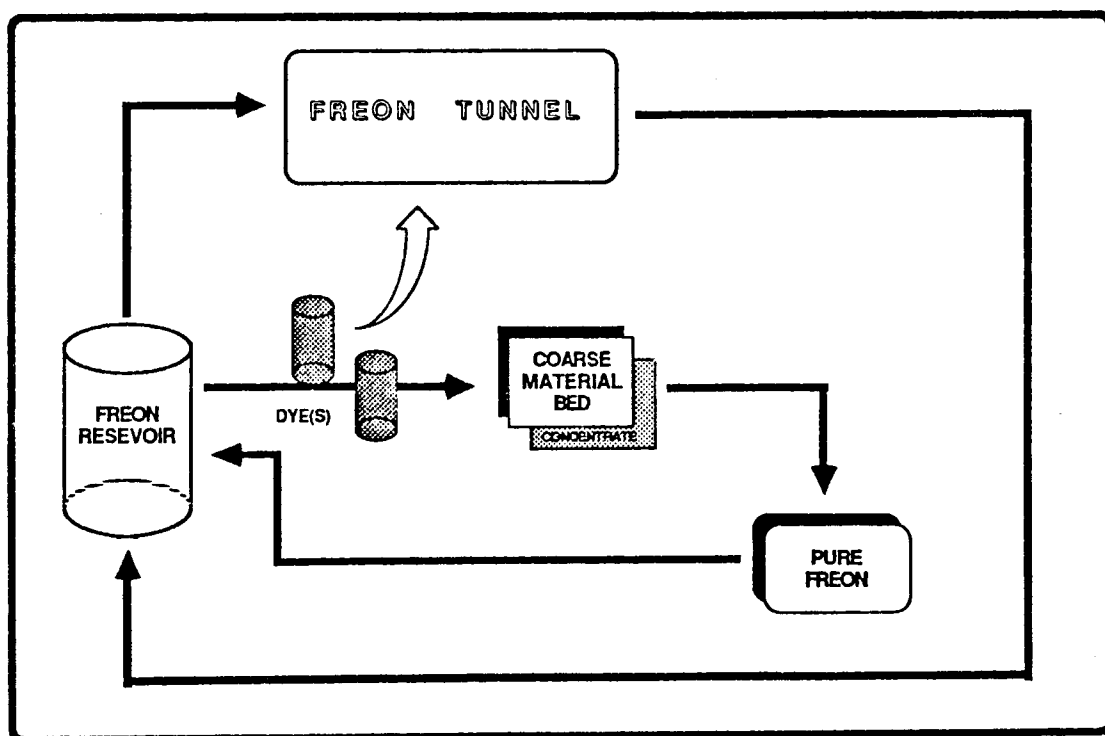


Figure 4.5. Coarse material bed separation system.

carbon to remove the dye is \$50 based on 1985 dollar value and conversations with personnel at RSE, a manufacturer of activated carbon.) It is anticipated that 20 grams of dye will be retained per pound of carbon.

3. DYE RETENTION ON FIBERS

Disperse dyes account for almost all of the dyeing of cellulose acetate and polyethylene terephthalate textile fibers. Therefore, it would be theoretically possible to remove the disperse dyes from the Freon by passing the contaminated Freon through pads of cellulose acetate. The dye would attach itself to the pads while allowing the Freon to pass through freely. When the fiber has become saturated with dye, it could be removed and replaced with another pad. Figure 4.6 illustrates this process. The contaminated Freon from the tunnel would travel through a holding tank layered with cellulose acetate pads. The

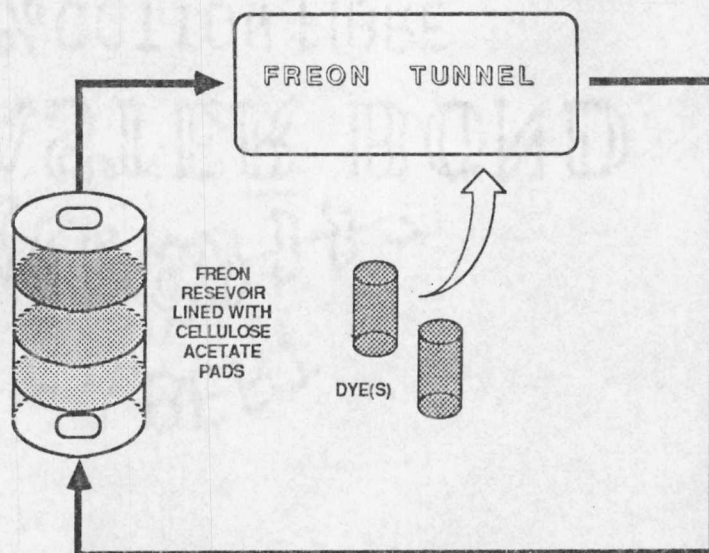


Figure 4.6. Dye extraction by utilization of cellulose acetate.

dye would remain in the holding tank in the fiber while the pure Freon traveled back to the tunnel for further use. The cellulose acetate pads may be purchased from Micro Filtration Systems (MFS), Dublin, California. The cost of the pads is dependent on the size required. (A 293 mm diameter pad from MFS would cost \$86 based on 1985 dollar value. It is unknown as to exactly how much dye the pads will absorb.) However, based on conversations with MFS personnel, it is anticipated that one 293 mm pad would retain approximately 7 grams of dye.

4. FREON DISTILLATION

It is theoretically possible to separate the dye from the Freon by distillation. The relatively low boiling point of the Freon as compared to that of the dye makes this a viable separation technique. A proposed Freon distillation system is illustrated in Figure 4.7. When the diffusion modeling has been completed, the contaminated Freon travels to the reservoir. From the reservoir the contaminated Freon will be sent batchwise to a distilling pot. The pot will be maintained at 60° C either through the use of steam or a conventional heating mantle. Under these conditions, the Freon would become a vapor and thus travel up the column (1.22 m) leaving the dye in the pot [10]. As the Freon vapor passes through the cooling coils, it will condense back to the liquid state. The pure Freon would then be collected in a holding tank and from there be pumped back to the reservoir for further use in the tunnel. (The cost of this distilling system is on the order of \$100,000 based on 1985 dollars. This cost analysis was performed by Glitsch Package Company, West Caldwell, New Jersey.)

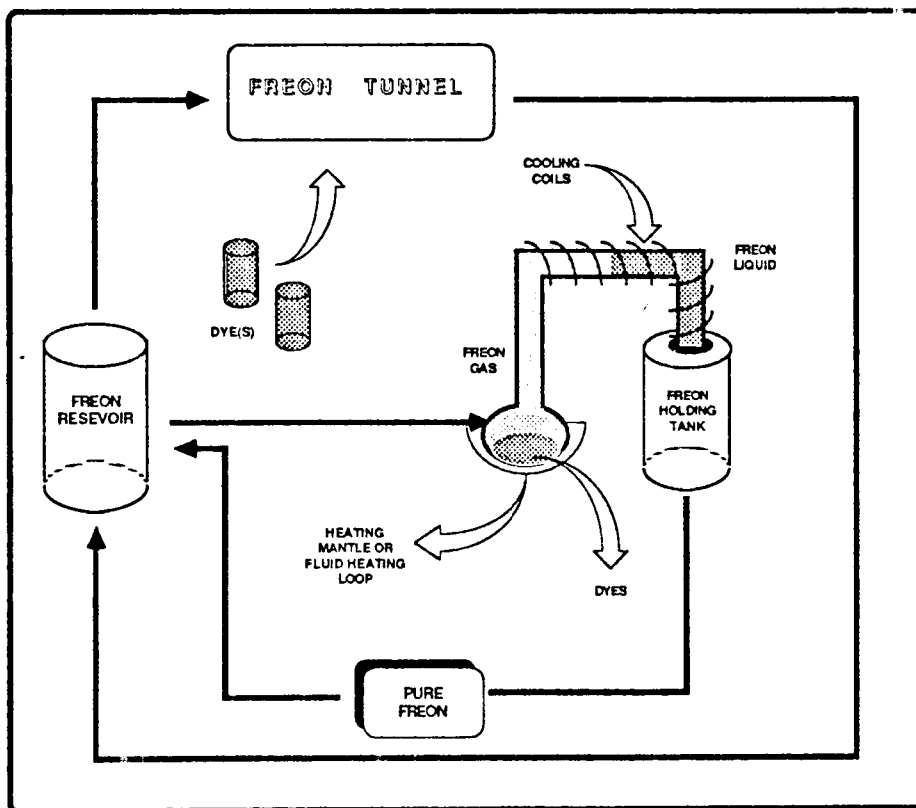


Figure 4.7. Separation by Freon distillation.

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

While several hundred dyes and classes of dyes were researched for suitability for diffusion modeling in a Freon atmospheric boundary layer simulation tunnel, only those dyes which met the established criteria were noted in Chapter I. Of those dyes found suitable for the use in the tunnel, it is recommended that disperse dyes be used in favor of solvent-soluble dyes. This is due to the fact that disperse dyes are anticipated to act, in the Freon, similar to a near-colloidal aqueous dispersion and this is felt to be more advantageous for diffusion modeling than solvent-soluble dyes which should "dissolve" in the Freon. It is suggested that anthraquinone-disperse dyes be used in favor of azo-disperse dyes since their light fastness grades are, in general, better than the azo class.

Chapter II provided a method for mixing dyes with various Freons to obtain lighter-than- and heavier-than-air spills. It was noted that the boiling point of Freon 113 is far above the boiling points of the other Freons identified. Thus, mixing of the dyes with various Freons could present a problem, coupled with the fact that the temperature in the tunnel should be below the boiling point of the Freon in the injection solution. It is anticipated that the Freon in the injection solution may vaporize if the temperature in the tunnel exceeds the boiling point of the injection solution.

Chapter III researches various separation systems for extracting the dye from the Freon after it has been used in the simulation.

Taking into consideration the properties of the dyes suitable for use in Chapter I and the cost and capabilities of the various separation techniques outlined in Chapter III, it is recommended that a coarse material bed be utilized. The use of a coarse material bed comprised of activated carbon would effectively filter the dye from the Freon. This separation process would not limit the use of any dye in the tunnel as in a cellulose acetate system, which would only be suitable for the filtration of disperse dyes. However, if only disperse dye were to be used in the tunnel, then the filtration using the cellulose acetate pads would be recommended. Both of these systems are more economical than a reverse osmosis unit or a Freon distillation column and should provide the degree of efficiency required.

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APPENDICES

APPENDIX A

LIGHT FASTNESS GRADING

Fastness is defined as the properties of dyed or printed textile materials in relation to the extent to which they are resistant to the various conditions which will destroy or remove the color. Such considerations may be approached from two angles:

1. In relation to the behavior of a colored article of commerce and having regard to consumer requirements.
2. In relation to the properties of individual dyes on specific substrates from which consideration a dyer or printer selects dyes having the required properties.

For the application defined herein, Item 2 will be employed. Thus, it is desirable to know the degree of light fastness, which is the degree of resistance shown by colored materials in the color-destroying influence of light. Two methods are used in fastness testing: (1) by exposure, under glass, to daylight and (2) by exposure in a fading lamp. Tests are usually carried out on a comparative basis by exposing unknown patterns against a series of dye standards representing the eight different grades (on the scale of 1 to 8).

Fading lamps are instruments designed to function as a greatly accelerated method of assessing the fastness to light of colored materials. Evaluations can be made after a few hours' exposure in such lamps which would take some weeks to obtain by exposure to daylight. Such lamps use various types of electrical illumination of high

intensity, and refinements are introduced to obtain a quality of light as close as possible to that of daylight [10].

The method of recording the assessments obtained after carrying out a fastness test is termed fastness grading. In the case of light fastness, the degree of resistance is expressed in terms of a 1 to 8 scale in which 1 represents the lowest fastness (i.e., poor) and 8 represents the highest fastness (i.e., complete resistance) while general fastness properties are expressed in terms of a 1 to 5 scale (1 is the lowest and 5 is the highest) [11]. It would not be possible to use a fading lamp to remove the dye because the dye only loses its color. The dye would still remain in the Freon and eventually contribute to altering the properties of the Freon.

APPENDIX B

DYE STRUCTURE AND PREPARATION

Auramine O, shown in Figure B.1, was first prepared in 1883 by Caro and Kern from 4,4'-bis(dimethylamino)benzo-phenone (Michler's Ketone) by heating it with ammonium chloride and zinc chloride at 150° to 160° C. A more recent method consists of passing ammonia over a mixture of 4,4'-bis(dimethylamino)diphenylmethane, sulphur, oxalic acid, and salt at a temperature which is gradually raised from 125° to 170° C and converting the resulting dye base into its hydrochloride [2].

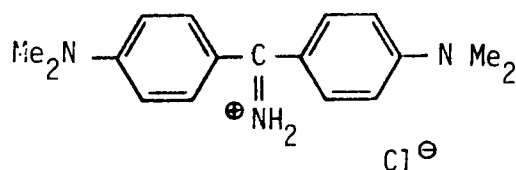


Figure B.1. Chemical structure of auramine.O.

Acridine orange, shown in Figure B.2, is manufactured from 4,4'-bis-(dimethylamino) diphenylmethane by dinitration, reduction to the diamine, cyclization to the dihydroacridine by heating with an acid, then oxidizing and converting into the zinc double chloride [2].

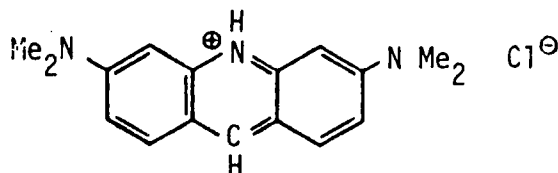


Figure B.2. Chemical structure of acridine orange.

Rhodamine B was placed on the market in 1887 by BASF. As shown in Figure B.3, it is obtained by condensing phthalic anhydride with m-diethylaminophenol; reaction takes place in two stages, the first giving the intermediate (a) and the second the dye base (b), which is converted to the dye (c) by acidification [2].

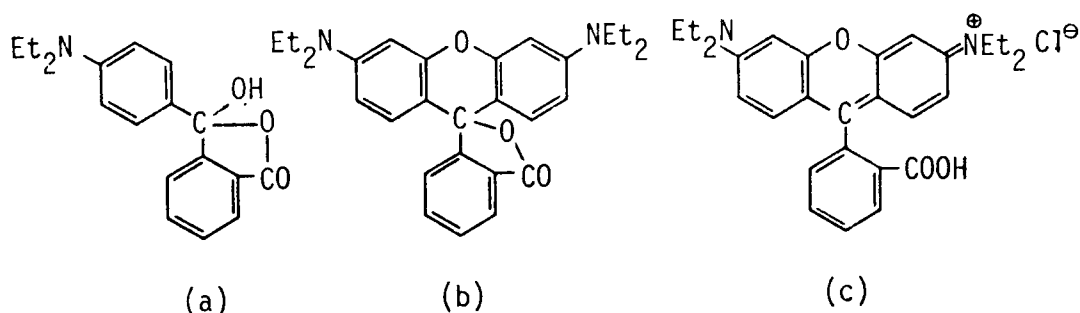


Figure B.3. Chemical structure of rhodamine B.

Coomassie violet 2R is a sulphonated derivative of the rhodamines (see Figure B.4). It is obtained by sulphonation of the rhodamines or by condensation of phthalic anhydride with m-chlorophenol, reaction of the resulting dichlorofluorane with a primary aromatic amine and sulphonation of the product [2].

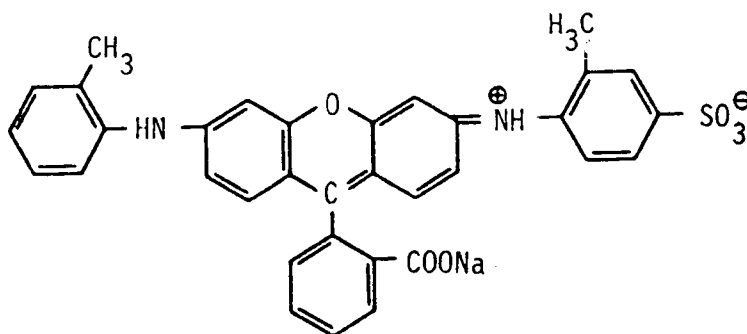
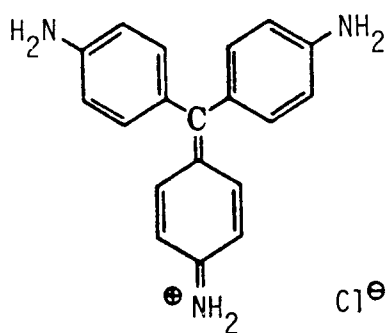
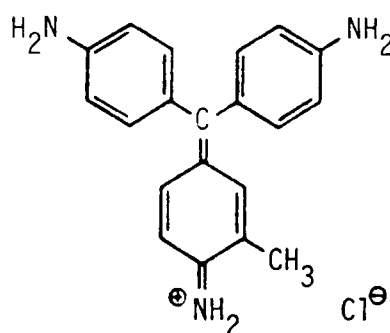


Figure B.4. Chemical structure of coomassie violet 2R.

Magenta is produced by oxidization of crude aniline with such oxidizing agents as mercuric chloride, arsenic acid, or nitrobenzene. Otto and Emily Fisher showed that it consists mainly of a mixture of pararosaniline and homorosaniline [7] (see Figure B.5).



(a) Pararosaniline



(b) Homorosaniline

Figure B.5. Chemical structure of magenta.

The compositions varied according to the amounts of o- and p-toluidine present in the crude aniline. Other reactions are used for producing rosanilines and some of the more important processes are described.

B.2. FORMALDEHYDE PROCESS

Formaldehyde reacts with aniline and aniline hydrochloride to give 4,4'-diamino-diphenyl-methane; this can be condensed further with aniline in the presence of iron filings and an oxidizing agent such as nitrobenzene, and on converting the product into its hydrochloride [2]. Figure B.6 illustrates this process.

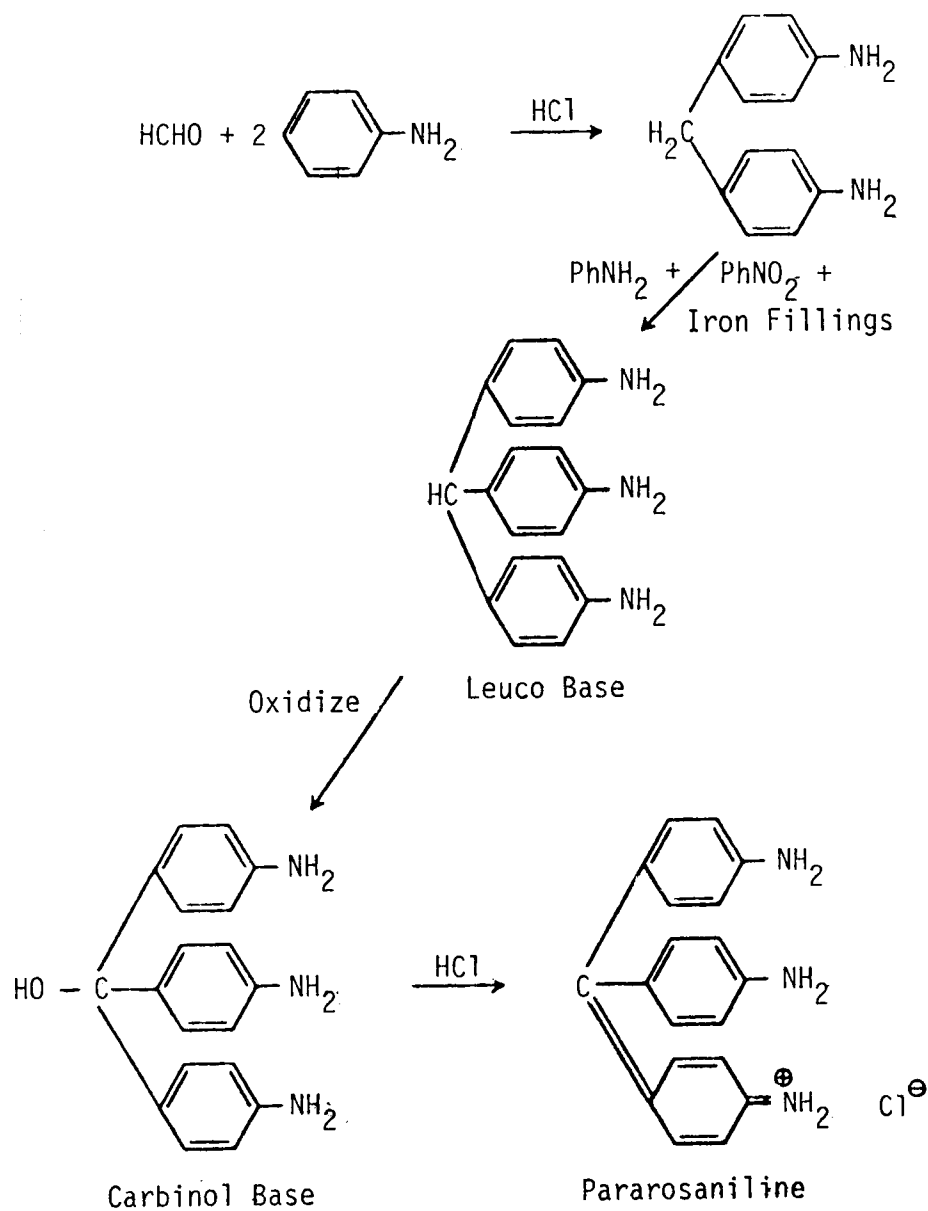


Figure B.6. Formaldehyde process.

B.2. PHOSGENE PROCESS

Reaction of N,N-diethylaniline with phosgene in the presence of zinc chloride yields, 4,4'-bis(dimethylamino)-benzophenone, and this may be condensed with a further molecule of dimethylaniline (or another base) to yield a rosaniline base [2]. Figure B.7 illustrates this process. The dye crystal violet is obtained in this manner.

Dyes of the methyl violet series are important methylated rosanilines with shades varying from reddish to bluish violet according to the degree of methylation. The redder brands can be made by methylation of rosaniline, but the bluer ones are made by oxidizing dimethylaniline with copper sulphate in the presence of common salt and phenol [7]. The series of reactions required to produce methyl violet is illustrated in Figure B.8.

Victoria blue B is a triamino derivative of naphthyl-diphenylmethane (see Figure B.9). It is obtained by condensing 4,4'-bis(dimethylamino)-benzophenone chloride with N-phenyl-B naphthylaniline, N-ethyl-naphthylamine, and N-methyl-N-methyl-Nphenyl-naphthylamine, respectively [2].

Flat blue Z is obtained by oxidizing a mixture of N,N-diethyl-p-phenylenediamine and naphthol in alkaline solution [2] (see Figure B.10).

Induline dyes are obtained by heating azo compounds, especially 4-aminoazobenzene with aniline and aniline hydrochloride (see Figure B.11). The produce is a mixture of axine bases of varying complexity, insoluble in water. The conditions used differ widely--higher

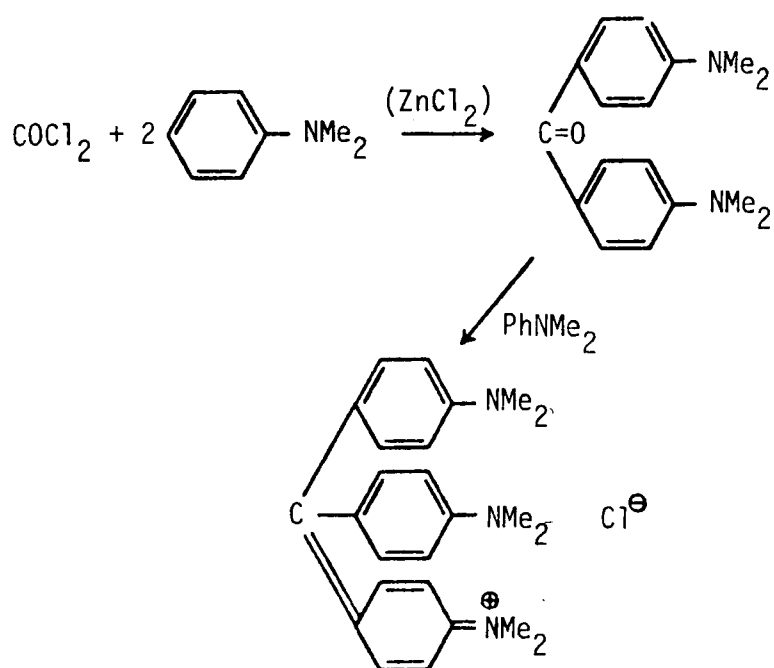


Figure B.7. Phosgene process.

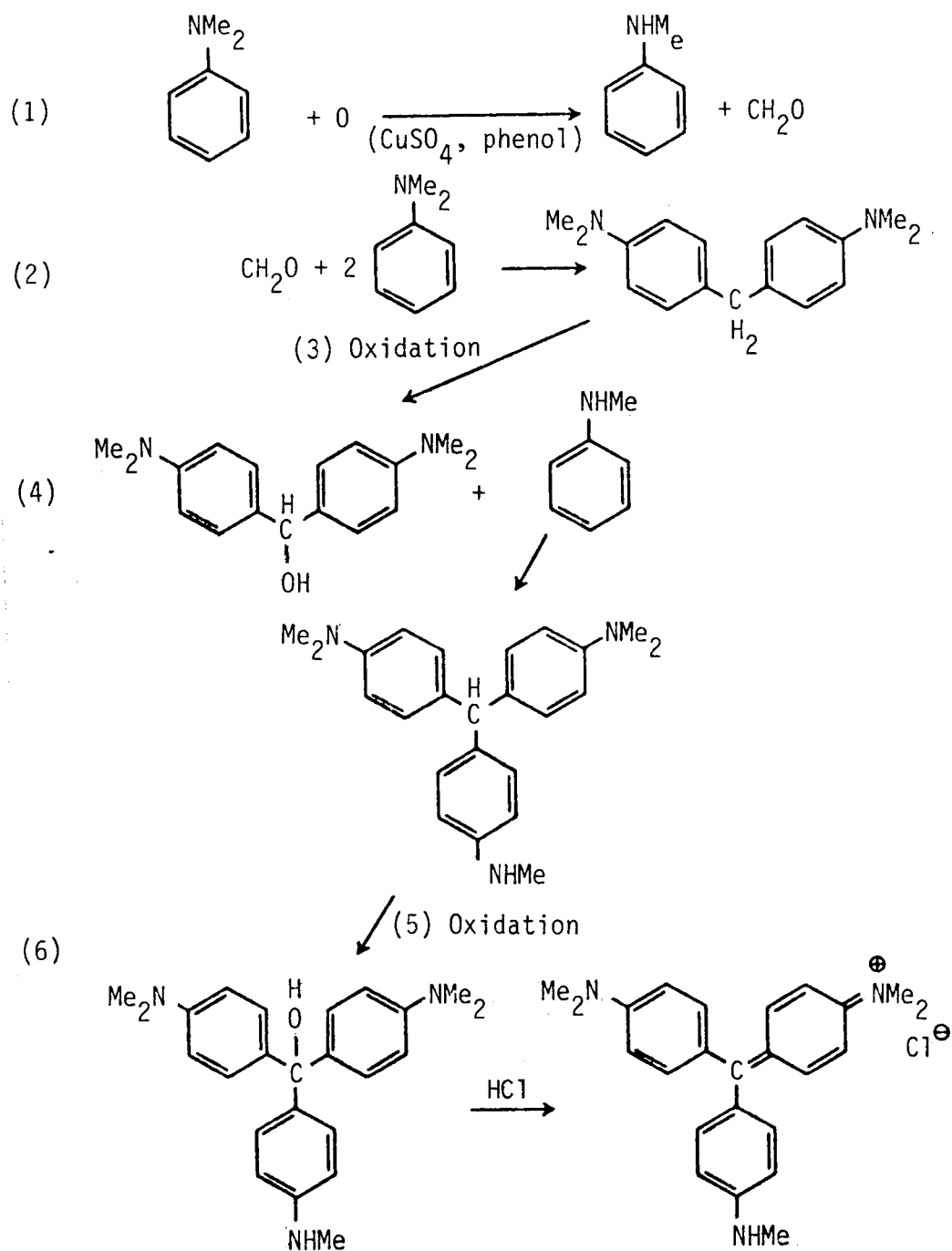


Figure B.8. Reactions to produce methyl violet.

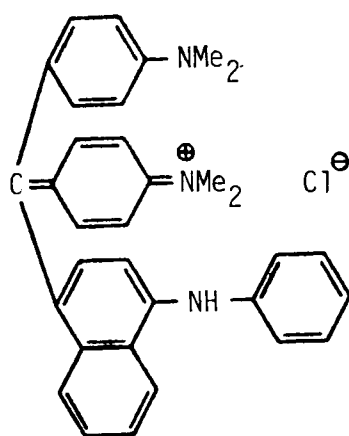


Figure B.9. Chemical structure of victoria blue B.

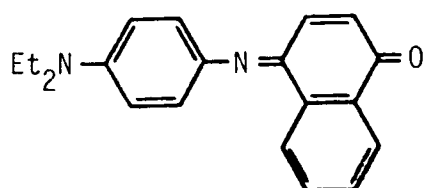
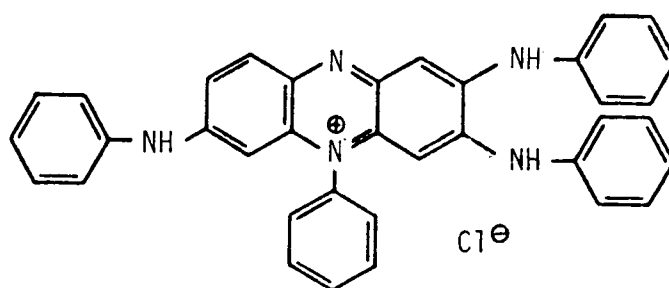
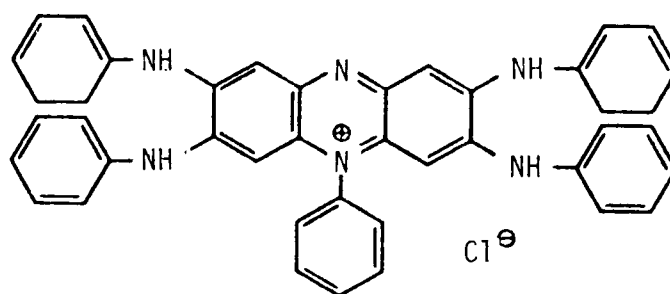


Figure B.10. Chemical structure of flat blue Z.



(a) Induline 3B



(b) Induline 6B

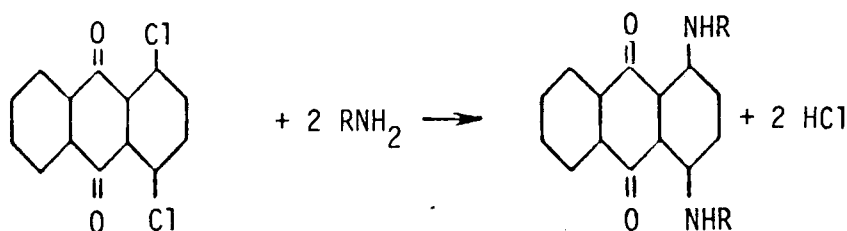
Figure B.11. Chemical structure of induline dyes.

temperatures and more prolonged heating being adopted when bluer shades are required [7].

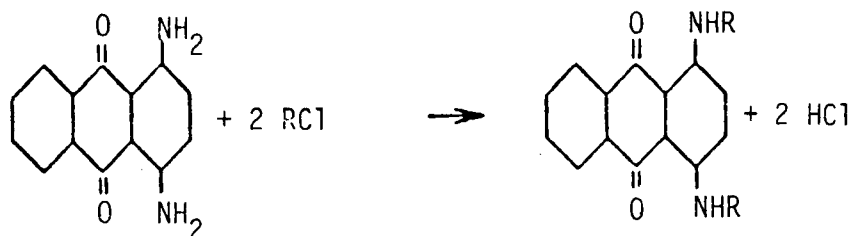
Nigrosine dyes are bluish-black to black products produced by the oxidation of aniline and aniline hydrochloride with nitrobenzene in the presence of ferric chloride at 160° to 180° C. The structure of the product obtained in the nigrosine is not definitely known. In 1938, Rudolf, a German scientist, concluded that nigrosine is a mixture of triphenazineoxazines (1,2) and phen-azineazines (3,4) plus 5 percent of induline 6B [7,2].

Azo dyes are characterized by presence in the molecule of one or more axo group-N-N-, which form bridges between organic residues, of which at least one is usually an aromatic nucleus. Many methods are available for preparing azo compounds, but manufacturing of axo dyes is based on the coupling of diazonium compounds with phenols, naphthols, arylamines, pyrazolones, or other suitable components to give hydroxyazo or aminoazo compounds or their tautomeric equivalents [12].

The 1,4-disubstituted aminoanthraquinone compounds can be obtained not only from the 1,4-dihydroxyanthraquinone but also from 1,4-dichloroanthraquinone with amines according to the reaction:



or from 1,4-diamionanthraquinones by reacting with arylhalides such that chlorobenzene, dichlorobenzene, or similar compounds [7]:



The main coloring in the 1,4-diaminoanthraquinone dyes is alizarin, present in the form of a glucoside known as ruberythric acid. Alizarin was obtained by acid hydrolysis of ruberythric acid (see Figure B.12), and it was identified as 1,2-dihydroxyanthraquinone by Graebe and Liebermann in 1868 [7]. In the following years, alizarin was prepared synthetically by Caro, Graebe, and Liebermann by fusion of anthraquinone-sulphonic acid with caustic soda [2].

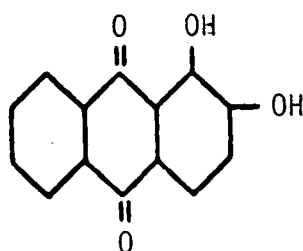


Figure B.12. Chemical structure of alizarin.

VITA

Karen M. Seiser was born in Canoga Park, California, on December 7, 1962. She attended elementary school in Canoga Park until June 1970; that same month her family moved to Huntsville, Alabama. She completed her secondary education in Huntsville and graduated from Grissom High School in June 1981. She entered The University of Alabama, Huntsville, in September 1981. In November 1983 she received a Bachelor of Science degree in Chemistry. In January 1984 she accepted a teaching assistantship at The University of Florida, Gainesville, and began study toward a Master of Science degree in Chemistry. In October 1984 she returned to Huntsville and began employment with Wyle Laboratories. In March 1985 she entered The University of Tennessee Space Institute, Tullahoma, and began study toward a Master's degree in Engineering Science while remaining employed with Wyle Laboratories.

The author is a member of the American Chemical Society and the scholastic honor societies Alpha Lambda Delta and Phi Kappa Phi. Ms. Seiser will pursue a doctorate in Engineering Science at The University of Tennessee Space Institute.