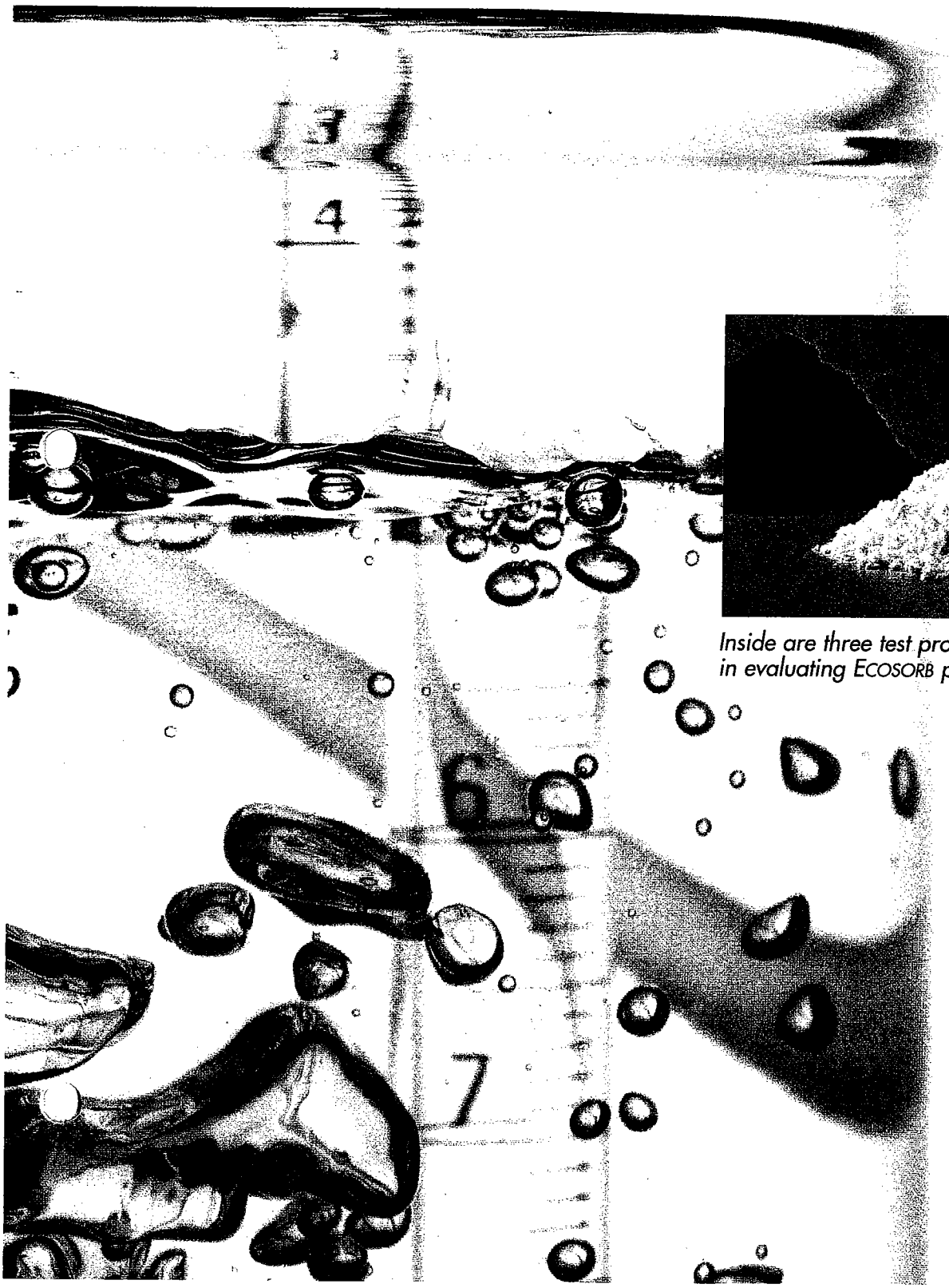
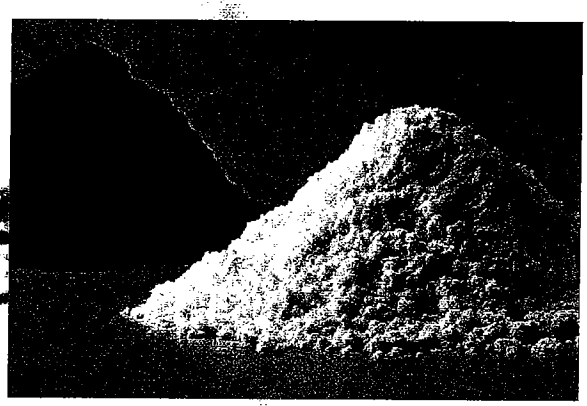
Graver Technologies

ECOSORB® Laboratory Evaluation Guide





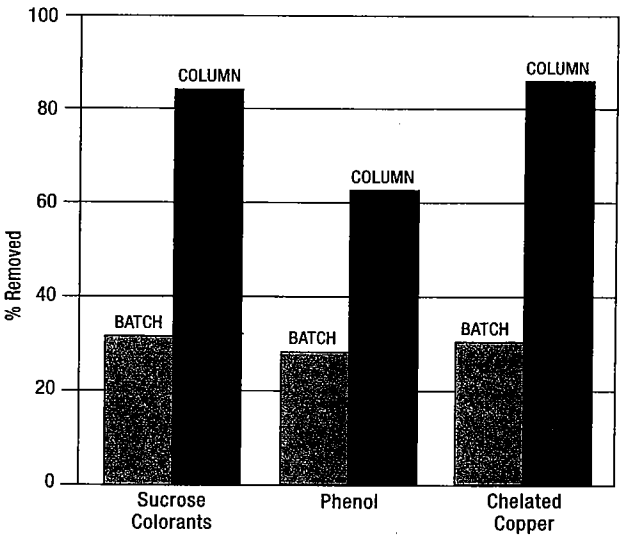
Inside are three test procedures to assist you in evaluating ECOSORB products

Introduction

Theory and experience tell us that adsorbents often work more efficiently in a column or precoat than in a batch contact. Important to the use of an adsorbent in a column or precoat are the kinetics and fluid hydraulics, which are largely controlled by the particle size. Smaller particle adsorbents offer more surface area and better kinetics but present a higher hydraulic pressure drop. These facts clearly indicate the need to balance kinetic and hydraulic requirements in adsorption processes.

ECOSORB products are a family of clean, easy-to-use fine particle adsorption media providing rapid kinetics at low operating pressures. Unlike other fine particle adsorbents and admixtures (e.g. mixtures of powdered activated carbon and diatomaceous earth), ECOSORB products use a patented process that reduces hydraulic pressure drop so effectively that they can be operated in industrial processes as precoats or in columns. Because precoat/columnar contact adsorption processes often work better than batch processes, ECOSORB products offer a unique means of achieving more efficient and higher purity adsorption processing. Figure 1 below shows three examples of the increased adsorption capacity provided by operating an adsorbent in a column versus a single batch contact.

FIGURE 1. Batch versus Column Efficiency (Carbon)



The following test procedures will assist researchers in evaluating ECOSORB products. The Adsorption Isotherm Test procedure provides a screening method for evaluating new adsorbents and identifying applications that will benefit from a columnar/precoat contact. The Column Test procedure and Precoat Filter Test procedure simulate typical methods used in industrial operation and are recommended for conducting process scale-up investigations.

Note: On page 7 of this bulletin is a chemical compatibility guide. Please consult this information before testing.

Adsorption Isotherm Test

Since many adsorption reactions are known to exhibit non-linear concentration dependent equilibrium we recommend using this procedure as the first step in all new investigations. In addition, adsorption isotherm tests are useful for investigating the effects of process variables (ph, ionic strength, temperature, dipole moment) on adsorption reactions.

Adsorption Isotherm (AI) Procedure

1. Place equal aliquots of the test fluid into each of three beakers. Place the filled beakers on magnetic stirrer/hot plate devices and initiate mixing. Adjust the temperature to the intended process temperature.

Note: At elevated temperature loosely cover beakers to prevent fluid loss via evaporation.

2. Weigh out each of the adsorbent (ECOSORB) dosages to be tested. One of the three test dosages should be the same as that used in the existing batch process. The other two dosages should be 5 to 10 times higher and 1/5 to 1/10 lower than used in the existing process. If an existing batch process dosage is unavailable use the following dosages for testing: 0.02, 0.10, 0.50% weight/weight. At convenient time intervals add the adsorbent(s) to the test fluid aliquots. It is important to stage the time between the dosages tested to allow for the subsequent removal of the adsorbent while maintaining a constant contact time for each dosage tested. At the end of the contact time the adsorbent is removed from the test fluid by filtration or centrifugation. For most adsorption processes a contact time of 30 minutes to an hour is adequate. If a new process is being investigated we recommend testing at more than one time interval to determine the appropriate contact time.
3. Using the appropriate analytical methods determine the concentration of adsorbate in the original sample and the adsorbent treated samples. From this information calculate the amount of adsorbate adsorbed per unit of adsorbent for each of the dosage levels tested. Prepare a

log-log base 10 graph of the results plotting residual concentration on the X axis versus adsorbate adsorbed per unit of adsorbent on the Y axis. Determine the slopes of the best fit straight line plot of the three dosage levels. The slope of the plot is a relative indication of the concentration dependence of a particular adsorption reaction. In general, the steeper the slope of the isotherm plot the higher the adsorption efficiency will be in a columnar or precoat process.

Table 1 and Figure 2 illustrate two adsorption isotherm plots and describe how to generate and graph the data. The first data set presents a case where the amount of adsorbate adsorbed per unit of adsorbent is directly proportional at all dosages. The second data set, however, shows a case where increasing the quantity of adsorbent does not results in a proportional increase in the adsorbate adsorbed. In cases like the second data set, column or precoat operation will often increase performance.

Note: There are several recognized methods for conducting adsorption isotherm testing. The methods described herein are based on the Freundlich method.

TABLE 1. ADSORPTION ISOTHERM TEST(S) EXAMPLES

DATA SET 1	Feed Concentration = 300 Units (A)		
(B) Adsorbent Dosage grams/liter	(C) Units of Residual Adsorbate	(D) Units Adsorbate Adsorbed	(E) Units Adsorbate Adsorbed per gram of Adsorbent
0.2	290	10	50
1.0	250	50	50
5.0	50	250	50
DATA SET 2	**Feed Concentration = 1000 Units (A)**		
0.2	600	400	2000
1.0	160	840	840
5.0	10	990	198

(A) = Feed concentration determined analytically.

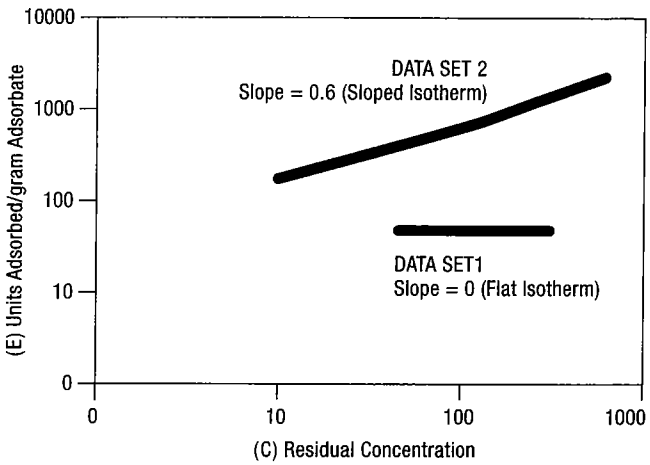
(B) = Adsorbent doses determined by researcher based on current process or reasonable estimate.

(C) = Concentration of adsorbate after treatment with adsorbent determined analytically.

(D) = Feed concentration minus (-) residual adsorbate concentration (i.e. A-C)

(E) = Units of adsorbate adsorbed divided by adsorbent dosage (i.e. D/B)

FIGURE 2. ADSORPTION ISOTHERM PLOTS



Column Test

Applications requiring the removal of trace contaminants, particularly those involving poorly adsorbed compounds, will perform best in a column. This is because columnar operation provides the equivalent of multiple stage contacts in a single unit operation. A number of common laboratory devices may be used to perform columnar adsorption testing — from a burette with a glass wool plug to a commercially available laboratory column. We recommend a column with a diameter of one inch or more and a length of at least six inches. The other basic requirements for testing in columns are a means of introducing the sample without disrupting the media bed; a device for controlling flow on the outlet of the column (e.g. a hose with a pinch clamp or a valve), and graduated containers for collecting samples. A diagram of a basic column apparatus is shown in Figure 3.

Column Test Procedure

1. Begin by making an ECOSORB slurry (~ 5% by weight) in a suitable solvent. (Suitable solvents should be chemically compatible with all components of the test system and miscible with the fluid being tested). Mix the slurry with a spatula or magnetic stirrer until it is free of clumps and air.
2. Pour the slurry slowly into the column and open the outlet valve to start flow and the formation of the Ecosorb media bed. Continue adding slurried ECOSORB until the desired bed depth is reached.

Note: Care should be taken not to allow the bed to run dry since this can cause cracks and fluid bypassing. For best results maintain the liquid level 2 inches above the top of the bed during formation and operation.

FIGURE 3. BASIC COLUMN APPARATUS

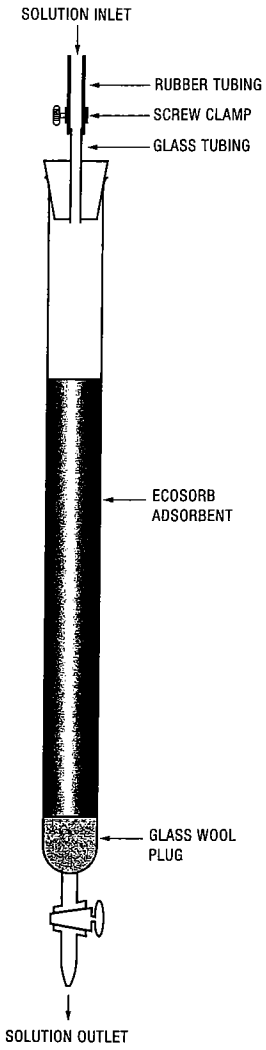
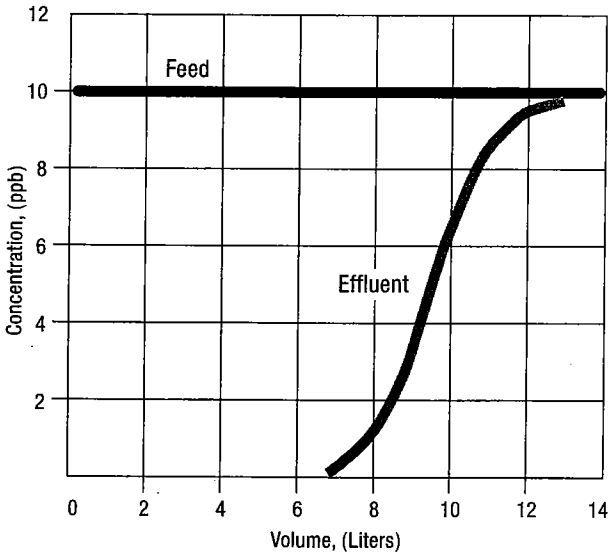


FIGURE 4. Example of a leakage curve plot



3. After the ECOSORB product has been charged to column, the bed depth should be measured and the amount of ECOSORB used noted. This information will be needed for scale-up calculations.
4. Intitate flow of the test fluid through the ECOSORB bed by gravity or pressure. As the test fluid is passed through the ECOSORB bed, samples of the effluent are collected at regular intervals and pressure drop and total throughput are recorded.
5. Using the appropriate analytical methods determine the concentration of the adsorbate in the original sample and the treated samples. Use the volume throughput and analytical data to construct a leakage curve where adsorbate leakage is plotted against volume throughput. This plot, along with the amount of adsorbent used and the bed volume, will provide the basic information needed for scale-up. Figure 4 is an example of a leakage curve plot.

Note: The test fluid being passed through a bed of ECOSORB should be generally free of suspended solids. Pre-filtration of the test fluid will help prevent fouling of the adsorbent surfaces and prevent uneven flow distribution across the bed.

Scale-Up Considerations

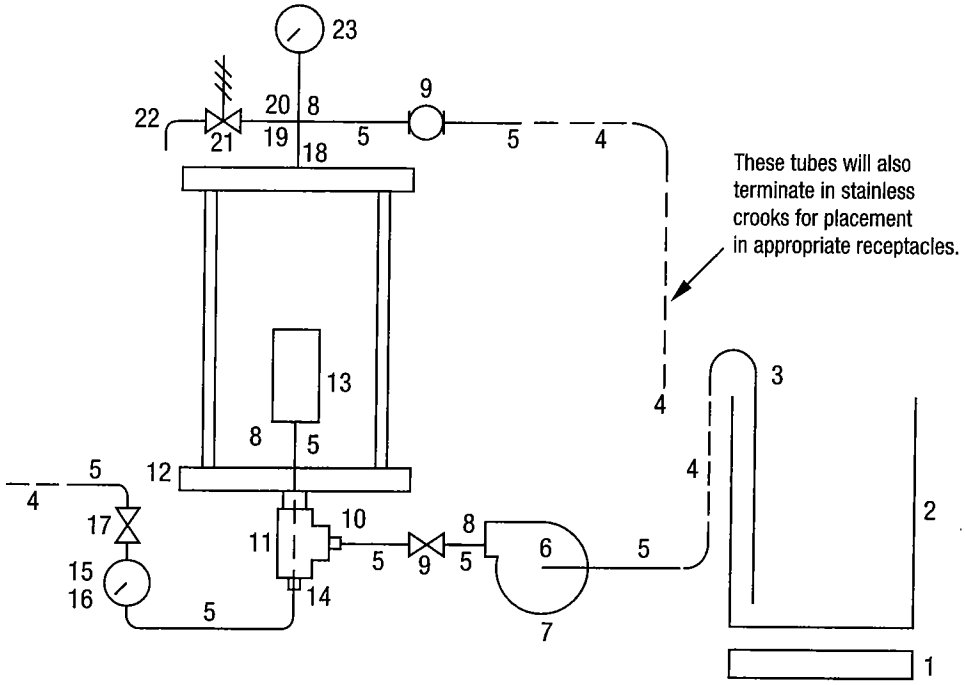
For laboratory data to be useful for scale-up purposes, the operating parameters of bed height, pressure drop, and flow rate per unit area should be very similar between lab and full scale. Broad extrapolation on the above parameters is not recommended due to the difficulty in predicting media compression effects.

Precoat Filter Test

This procedure allows for the evaluation of ECOSORB products in industrial polishing applications employing leaf or plate and frame filters. This test will be particularly useful to investigators comparing the performance of an ECOSORB product to that of powdered carbon and filter aid. The procedure below is written for the Graver Technologies Laboratory Precoatable Filter system (LPF).

Caution: The Laboratory Precoatable Filter is designed for use with aqueous solutions only. A schematic of this system is shown in Figure 5.

FIGURE 5. GRAVER TECHNOLOGIES LABORATORY PRECOAT FILTER SYSTEM



FITTING LIST

1. Hotplate
2. Beaker
3. S.S. Tube
4. Tygon Tubing
5. S.S. Tube
6. Adapter
7. Pump
8. Adapter
9. Ball Valve
10. Adapter
11. Street Tee
12. Vessel
13. Septum
14. Adapter
15. Pressure Gauge
16. Tee
17. Needle Valve
18. Bushing
19. Nipple
20. Cross
21. Relief Valve
22. Street Elbow
23. Pressure Gauge

For laboratory and full scale we recommend using Ecosorb in combination with a filter aid such as diatomaceous earth. The filter aid is used to form a precoat underlayer on which the Ecosorb is precoated as a second layer. The filter aid used for the underlayer should be capable of providing the level of filtration required by the application (i.e., removing turbidity and microorganisms).

Lab Precoat Filter (LPF) Test Procedure

Caution: Read operating and safety instructions herein before attempting to use Graver Technologies Laboratory Precoat Filter System.

1. Weigh out filter aid underlay and ECOSORB sample for testing according to table 2. See steps 3 and 4 for recommended dosages.

TABLE 2

Filter Aid Dosage lbs/ft²	Filter Aid Dosage Kg/m²	Ecosorb Dosage (As Is) Lbs./ft²	Ecosorb Dosage (As Is) Kg/m²	LPF Unit Filter Aid Dosage Grams	LPF Unit Ecosorb* Dosage (As Is) Grams
0.15	0.732	.375	1.83	2.6	6.5
0.25	1.220	.625	3.05	4.3	10.8
0.35	1.710	.875	4.27	6.0	15.1
0.45	2.200	1.125	5.50	7.8	19.5

** Standard 60% moisture product*
Note: LPF area = 0.038 ft² (0.0035 m²)

2. Put twelve hundred (1,200) milliliters of clean precoating fluid into the precoat/supply beaker. Place the magnetic stirring bar into the precoat/feed beaker and initiate moderate stirring. If the test is to be performed at an elevated temperature, heat and maintain the precoating fluid using the hotplate (do not exceed 80 degrees Centigrade). With the isolation, bypass and throttling valve fully open, start the system's pump. Next partially close the filtrate throttling valve to displace the air from the filter vessel and connecting lines. After the LPF system is completely filled, partially close the bypass valve and open the filtrate throttling valve to establish a filtrate flow of 200 to 300 ml per minute.
3. After establishing the recirculation conditions in step 2 add filter aid (e.g. diatomaceous earth) to the precoat beaker/fluid. The amount is usually selected to give 0.15 to 0.35 lbs of filter aid/ft² of filter area (0.73 to 1.71 kg of filter aid/m² of filter area). (See table 2 for LPF dosage in grams). Allow the fluid to recycle until an even layer of filter aid has deposited on the LPF filter surface and the precoating fluid has cleared. (Do not exceed 20 psig on pressure gauge P1 while precoating the filter aid.)

4. Continue the precoating procedure using the same recirculation conditions established in step 2. Add preweighed amount of ECOSORB to the precoat/supply tank. Allow the fluid to recirculate until the ECOSORB precoat has formed on top of the filter aid and the precoating fluid has cleared. Typically the ECOSORB is precoated to give 0.625 to 1.125 lbs (as is) of ECOSORB/ft^2 of filter area (3.05 to 5.50 kg/m^2 of filter area). (See table 2 for LPF dosage in grams). (Do not exceed 30 psig on pressure gauge P1 while precoating ECOSORB precoat.)

Note: To maintain the integrity of precoat layers applied in steps 1 through 4 maintain pumping throughout course of test. Turning off the pump will crack precoat layers and cause fluid channeling.

5. Fill the precoat supply beaker with the material to be processed. There will be a small amount of the precoat fluid in the LPF housing and beaker. If the fluid is similar to or the same as the process fluid, proceed to step 6. If it is desirable to minimize the amount of precoat fluid in the effluent it should be displaced by the feed material before proceeding to step 6.
6. Adjust the filtrate flow rate to the desired processing rate by partially closing the filtrate throttling valve. Measure filtrate rate with a graduate cylinder and adjust accordingly until the desired rate is established. See Table 3.

TABLE 3

Process Flow Rate		Equiv. flow in LPF Unit
gallons/min./ft^2	liters/min./m^2	milliliters/minute
0.05	2.03	7.2
0.1	4.06	14.4
0.2	8.13	28.8

7. Begin collecting filtrate samples at regular intervals (normally every 0.5 to 1.0 hours) to construct a throughput versus leakage curve. At each interval, record the throughput and pressure. Over the course of the experiment pressure drop across the precoat will increase and it will be necessary to begin opening the filtrate throttling valve and later to partially close the bypass valve to maintain the target flow rate. When the target flow rate can not be maintained at a pressure drop of 40 psig (P2-P1) discontinue the test.

Caution: The LPF housing is rated @ 50 psig max. Pressures above 50 psig can crack the housing and cause fluid to leak out. The pressure relief valve protects both the unit and operator should the pressure accidentally exceed the maximum for any reason. The valve should be set to relieve the pressure at 40-45 psig.

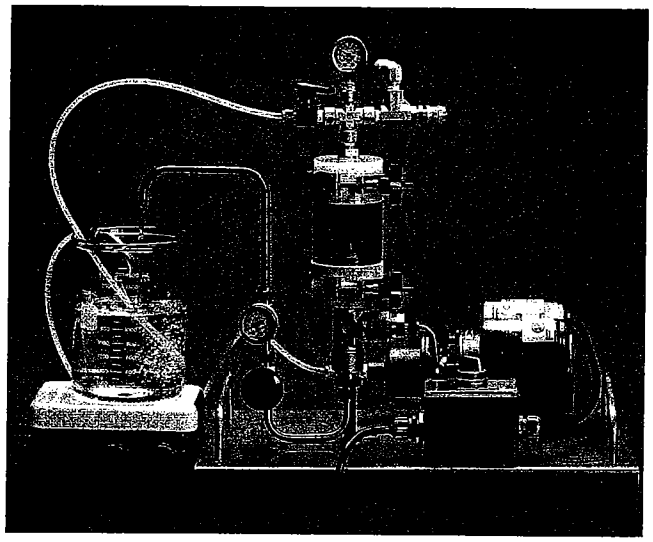
SETTING THE VALVE: Loosen the lock nut by turning it counter-clockwise. Apply 40-45 psig of water pressure to the housing. If the valve is leaking slowly turn the tension adjuster clockwise until it stops. If the valve is not leaking slowly turn the tension adjuster counter-clockwise until it just begins to leak. Tighten the lock nut to prevent an inadvertent change in the setting.

Note: For applications where decolorization/purification capacity from a precoat is not sufficient, we recommend reducing the unit area flow rate and/or using a combination of an admixture and precoat. For further suggestions on difficult applications contact Graver Technologies.

Scale-Up Considerations

Data from an LPF filter can be extrapolated directly to the performance of an industrial filter on a flow rate per unit area and dosage per volume processed basis.

PRECOATABLE FILTER (LPF) UNIT



Graver Technologies leases and sells Laboratory Precoatable Filter (LPF) units (shown above). For technical assistance and/or information about the Graver Technologies Laboratory Precoatable Filter System contact:
Graver Technologies phone: 1-800-249-1990
or fax: 302-731-1707.

Chemical Compatibility Guide

ECOSORB products are generally suitable for applications at pH values between 2 and 12. Standard ECOSORB products are not suitable for use with strong acid, strong base, or with strong oxidizing agents.

Caution: Low moisture Ecosorb material such as C-903 can produce significant amounts of heat when in contact with certain solvents (eg., freon). The process must include provisions for dealing with the exotherm.

Note: The lab precoat unit is designed for use with aqueous systems only. Do not use with solvents as they may damage the unit and/or cause injury.

The following solvents have been tested and found to be compatible with use of standard ECOSORB products. For questions regarding other solvents see the ECOSORB Product Selection Guide or contact Graver Technologies.

- **Water**
- **Methanol**
- **Ethanol**
- **2-Propanol**
- **Tetrahydrofuran**
- **Acetonitrile**

For technical assistance contact us by
Phone: 1-800 249-1990
Fax: 302-731-1707
E-mail: ecosorb@gravertech.com

Graver Technologies

200 Lake Drive,
Glasgow, DE 19702 U.S.A.

302- 731-1700
Fax 302-731-1707

e-mail: ecosorb@gravertech.com
web site: www.gravertech.com

ISO 9001 Registered
Quality Committed

G-181 05/00 Printed in U.S.A.