MICROCOPY RESOLUTION TEST CHART
DEPARTMENT OF THE ARMY
PINE BLUFF ARSENAL
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TECHNICAL REPORT SMCPB-TR-29

IN-PLANT REUSE OF POLLUTION ABATED WATERS

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This task was performed at Pine Bluff Arsenal, Arkansas, as part of a US Army-wide omnibus project.

-Granular Carbon Treatment
-Carbon Regeneration
-Carbon Disposal
-Economic Analysis
-Total Organic Carbon (TOC)

A Carbon Column Pilot Treatment Facility is described which will be evaluated during the next several years to treat a portion of PBA wastewater to determine the level of savings which may be realized from improved treatment; reduction of total organic carbon (TOC); better control of aquatic toxicants; removal of EPA listed chemical compounds; and reduction of sludge for ultimate disposal. The addition of new waste streams to the PBA facility and the attendant EPA requirements for ultimate disposal of sludge and hazardous waste made the task a timely study.
Item 19, cont'd

- *Lepomis macrochirus*
- Sludge Dewatering
- Lamella Gravity Settler
- Parkson Sludge Press
- Continuous Monitor (TOC)
- Hydrocyclones

Item 20, ABSTRACT, cont'd

The NMT Task resulted in installation of a Pilot Carbon Column Treatment Plant; identified two sources of commercial granular carbon, three methods for carbon disposal; three methods for removal of sludge from the liquid phase; an Economic Analysis for carbon disposal vs regeneration; an Economic Analysis for a full-scale granular Carbon Treatment Plant (5.2-7 year payment) and installed a process control system. Attendant commercial data is reported.

Since PBA does not operate all plants on an annual basis or at the same time or mode, it will take several years to evaluate the effectiveness of the system to meet effluent quality guidelines and accomplish the projected gains.
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1.0 ACKNOWLEDGEMENTS: The authors wish to recognize the contributions of the PBA Environmental Management Office - Mr. Wendell Fortner and Mrs. Lula Dickson; the Quality Assurance Laboratory - Mr. Joe Castagno; the Directorate for Facilities Engineering for electrical and civil engineering support; the Plants/Materials Engineering Division of the Directorate for Engineering and Technology for electronics support - Mr. Huey DeSoto; Capt. James Stinnett for carbon economic analysis and Mr. Larry Stosky, Project Manager at Picatinny Arsenal. The engineering literature provided by the several process equipment companies was also appreciated, along with the Biological Testing Protocols provided by a contractor, Battelle Columbus Laboratories - Dr. Berry Goss. Mr. William McDonald and Mrs. Kathy Boyd contributed many hours to proofing and input to the word processor for which we are greatful.
2.0 **SUMMARY:** A Carbon Column Pilot Treatment Facility is described which is being evaluated during the next several years to treat a portion of PBA wastewater to determine the level of savings which may be realized from improved treatment; reduction of total organic carbon (TOC); better control of aquatic toxicants; removal of EPA listed chemical compounds; and reduction of sludge for ultimate disposal. The addition of new waste streams to the PBA facility and the attendant EPA requirement for ultimate disposal of sludge and hazardous waste made the Task a timely study.

The MMT task resulted in installation of a Pilot Carbon Column Treatment Plant; identified two sources of commercial granular carbon, three methods for carbon disposal, three methods for removal of sludge from the liquid phase; an Economic Analysis for carbon disposal vs regeneration; an Economic Analysis for a full-scale granular Carbon Treatment Plant (5.2-7 year payment) and installed a process control system. Attendant commercial data is reported.

Since PBA does not operate all plants on an annual basis or at the same time or mode, it will take several years to evaluate the effectiveness of the system to meet effluent quality guidelines and accomplish the projected gains.
3.0 INTRODUCTION/OBJECTIVE: PBA is a Department of Defense AMCOM managed facility with a highly varied pyrotechnic chemical mission. Within the PBA manufacturing complex, several wastewater treatment facilities were built to treat wastewater from various plants. This task deals with improvements for the existing Central Wastewater Treatment Plant and treatment of liquid waste from a variety of industrial plants operated in various modes all of which discharge to the Central Treatment Facility (Appendix K-1). The plant is permitted (Appendix I-1) by EPA and consists of a lime/alum/polyfloc/powdered carbon treatment system wherein floc is discharged to a lagoon to settle and contain the sludge. The supernatant liquid is pH adjusted and discharged to a stream that enters the Arkansas River some two miles distant, which is a high flow navigable river. With time, several attendant problems are developing which this program addresses. These problems are: final disposition of large volume of sludge; better utilization or reuse of chemicals; better utilization of carbon to remove Total Organic Carbon (TOC), toxic chemical control (aquatic), and removal of organic compounds which may be added to the EPA list (Hexachloroethane) in the not too distant future; ability to meet more restrictive environmental laws; and the ability to treat new plant waste streams being added at PBA.

The project Scope of Work is reported in Appendix A, with Appendix B reporting the funding distribution for the $160K project.
4.1 Operating Concept for overall Wastewater Treatment Facility (APPENDIX C)

Process waste water enters the Central Waste Treatment Facility (CWTF) in the first chamber of the treatment sump. During existing operation powdered carbon is added first in the treatment sump (See Appendix G) using a chemical feeder system (See Appendix D-11). When the granular activated carbon columns (Appendix D-4) are in service, the powdered carbon will not be added. Alum and lime are added next to coagulate the floculatable material. The waste water feed to the sump is adjusted to a pH of approximately 6 with alum, and then to a pH of 11 with lime. This treatment causes floculation. Presently this floculated process water is pumped to the lagoon where sedimentation of the floc occurs. The lagoon gives enough retention time for the floc to settle before the pH is adjusted and the clarified water is discharged. The elimination of the powdered carbon addition will dramatically reduce the volume of sludge accumulation in the lagoon (Appendix J-1). During Pilot Plant Operation the waste water containing the floculate material is pumped (Appendix D-8) to the Lamella Settle (Center Vessel-Appendix D-2, Appendix K-3) for separation. The lamella settler contains diagonal baffles, or lamella, which give more surface area for separation. The solids precipitate to the bottom while the clarified water overflows from the top. Sludge is pumped off the bottom of the settler and the supernatant overflows to the carbon column feed tank. This supernatant, which has had the floculated material removed, is pumped through the prefilters to the carbon columns. The sludge is pumped to the Parkson Dewatering Press to be dewatered. The prefilters (Appendix D-9) remove any suspended floc which was not removed by the settler. Some of the feed to the columns can bypass the columns since the output from the carbon column feed pump is greater than the allowable flow through the columns.

Process wastewater flows through the columns in series. Water flows through column A first and then through column B (Appendix C). A bypass is installed so that flow may be directed only to column A. The treated water is returned to the last chamber of the sump. The discharge from the sump is pumped to the lagoon (Appendix D-7).

As implied above, the reduction of sludge volume will be accomplished using the sludge dewatering press as purchased from the Parkson Corporation (APPENDIX K-2). Organic polyelectrolytes will be added to the concentrated floc to enhance the efficiency of the dewatering press. The dewatered sludge from the press will be accumulated in sludge hoppers for proper disposal.
4.2 Operating Concept for Granular Carbon Treatment Facility (Appendix D-1, H-1, and G-1)

Five distinct operating modes are involved in running the Granular Carbon Treatment Facility (GCTF). They are: 1) Fresh Carbon Feed; 2) Wastewater Feed; 3) Surface Wash; 4) Backwash and 5) Spent Carbon Removal. These five operations are controlled using the central control panel (See Appendix D-3). This panel displays a simplified flow diagram for ease of operation. Lights indicate which valves are open so that flow may easily be verified. The valves in the carbon of the GCTF are air operated and explosion proof (See Appendix D-5) due to the potentially hazardous atmosphere caused by powdered carbon.

The first of these operation modes is Fresh Carbon Feed. Fresh carbon is loaded into the columns using the carbon feed system (Appendix D-1). The carbon feed system is composed of a hopper/eductor (Appendix H-2) and the necessary piping and valves to direct the carbon to the proper column (Appendix D-4). Granular activated carbon (8X30 Mesh) is poured from the bags in which it is supplied into the hopper of the hopper/eductor. Potable water is used for the motive force to slurry the carbon through the eductor of the hopper/eductor to the carbon columns. The water that is used to slurry the carbon exits the columns through a manifold which retains the carbon. A homogeneous bed of carbon is thus produced.

The second operation mode is Wastewater Feed. This is the main function of the Pilot Plant. Process wastewater flows through the columns in series. Water flows through column A first and then through Column B (Appendix C). A normally closed bypass is installed so that flow may be directed only to column A. The wastewater enters and exits the columns through manifolds so that the carbon bed is kept intact. Flow in the carbon column Pilot Plant is monitored with flow gages (Appendix C). One flow gage measures the total flow. The total flow is split into two streams: the column feed stream and the bypass. Flow is monitored with flow gages in both the column feed stream and the bypass. The total flow is monitored and recorded with a flow meter.

Due to the nature of the production operations, tremendous swings in both the volume of water and concentration of contaminants in the wastewater are observed. Because of these swings, analyses must be made to determine the CWTF influent loading so that proper treatment of the wastewater may be made. Analyses Appendix E-1 and E-2 must be made of the effluent water to ascertain that proper treatment was made and that the effluent conforms to the NPDES permit standards (Appendix I). Two analytical techniques are used to determine the quality of the
untreated and treated water. The analytical technique TOC (Total Organic Carbon) (Appendix E-1) is used to determine the presence of carbon containing organics. The analytical technique Conductivity is used to determine the presence of ions in solution. Both TOC and conductivity are monitored before and after the carbon columns. Thus proper treatment can be made and the effectiveness of the columns can be determined. When the quality of the effluent is not acceptable, then the carbon must be replaced.

The prefilters (See Appendixes C and D-9) remove any suspended precipitate that may overflow the settler. Only one prefiler is used at a time. When one prefiler develops an excessive pressure drop, which indicates that it has accumulated some precipitate, it is removed from service and the other prefiler is put in service. The spent prefiler is backwashed to prepare it to return to service.

The third operation mode is Surface Wash (See Appendix D-1). The surface wash was designed to dilute and wash down any floc that accumulates on the top of the bed. For the Surface Wash operation, potable water is injected into the column. This extra water then flows through the column like the wastewater. If the prefilters are working properly, it may never be necessary to use the surface wash.

The fourth operating mode is Backwash. Pressure gages are installed to determine the total pressure drop across the columns (See Appendix C). When a carbon bed develops a high pressure drop this means that the carbon has become compacted from the normal downflow operation of the column. When this happens the wastewater feed is shut off. Fresh water is flowed backward (See Appendix C) up the bed to lift the bed and reduce the pressure drop. This backwash flow exits the columns and flows to the drain.

The fifth operating mode is Spent Carbon Removal. Carbon is removed from the columns using the carbon removal system (See Appendix D-1). The carbon removal system is composed of eductors (See Appendix D-5), the spent carbon receiving tank (See Appendix D-10), and the necessary piping. An eductor is installed on each column to remove spent carbon. Potable water is used to slurri the spent carbon. The spent carbon is slurried to the spent carbon receiving tank where the water is drained from it or it can be transferred to a truck to be taken to a regeneration facility for regeneration (See Appendix J).
4.3 Activated Carbon Bench Studies and Commercial Data

Several experiments have been run on granular activated carbon to select the proper carbon for the carbon columns and to determine the carbon loading (See Adsorption Isotherm Method - Appendix G-2.). Adsorption isotherms were run on three brands of carbon: Filtrasorb 300/400 (Appendix F-1), Nuchor (Appendix F-2), and ICI. From these experiments the Nuchor and Filtrasorb carbons were determined to be equally effective for treatment of the wastewater at PBA, and more efficient than the ICI carbon. The Calgon carbon was selected based on price.

Work is still being conducted to determine the loading of the carbon relative to composition of various plant wates. The loading is the amount of adsorbate that is adsorbed per unit weight of carbon. Loading is being determined by flowing wastewater through a 5" diameter column (Appendix D-6) that is 6" deep by the following method. Waste water was obtained from the Central Waste Treatment Facility (CWTF) in 5 gallon containers. It was combined in a 50 gal tank. The waste water was floculated using alum and lime as it is done at CWTF. Alum was added to pH 5.5 and lime was added to pH 10.5. No carbon was added. The floc was allowed to settle and then was drained off the bottom of the 50 gallon tank.

The clarified waste water was then pumped carefully into the carbon column. The flow rate was established by timing the rate of the meniscus down the column. A valve at the discharge of the column was used to vary the flow rate. For the first run the flow rate was 1" of column per minute or 0.6 gpm/ft².

A 4 liter graduated cylinder was used to catch the effluent. Samples were taken every 2 liters. The samples were caught in screwed top test tubes. A sample valve at the base of the column was used to catch the sample. The sample line was flushed before catching the sample. The samples were analyzed for TOC by the water lab to determine break point which is the basis for carbon loading rates reported in Appendix J.
4.4 Evaluation of Carbon Treatment and Disposal Systems

The existing CWTF utilizes powdered carbon treatment primarily for removal of color in the PBA wastewater. The powdered carbon is added as a slurry in a sequential treatment process that also includes alum, lime and polyelectrolyte flocculation. The carbon ends up in the settled waste sludge, thus adding to the sludge volume. Current operation settles the sludge in a lagoon. In the future, final disposition will involve dewatering this sludge for deposit in a RCRA-approved landfill. Incentives for improving the carbon treatment include:

Reduce sludge volume to reduce landfill costs by eliminating carbon from the sludge.

Reduce carbon consumption by using granular carbon in a column vs powdered carbon in a batch process.

Insure adequate treatment with varying hydraulic loads.

Reduce labor costs associated with feeding powdered carbon.

Carbon treatment is typically employed to remove soluble organic matter from wastewater. Dyes from the colored smoke process at PBA are removed and effluent color is the primary guideline used in operation of the existing powdered feed system at the CWTF. Carbon feed is increased during episodes of high color. A potentially more serious problem is the presence of hexachloroethane (HEX), arising from the manufacture of HC smoke mix. Although colorless in solution, this compound is a RCRA-listed hazardous waste (Title 40, Code of Federal Regulations, Part 261, Appendix VIII). Groundwater contamination by this compound above background levels requires immediate corrective action. In addition, EPA may institute discharge standards for this particular compound in the future. A need for improved treatment is foreseen since the existing powdered carbon system has no means of automatically increasing treatment level. The CWTF operator must manually sample and visually test for color, then manually increase carbon rate. No test is made at the treatment plant. Assays are made upon discharge from the Settling Lagoon. Appendixes J-1 and J-2 report carbon consumption and loadings.

Granular carbon treatment in fixed bed adsorbers is known to afford a variety of benefits over powdered carbon treatment via slurry contacting. These benefits include:

Higher loading of waste per unit weight of carbon, thus reducing quantity of carbon used.

Granular carbon can be regenerated for reuse. Wastes are incinerated to harmless compounds during the regeneration
Granular carbon systems are normally operated with a minimum of two columns in series, such that the majority of waste removal occurs on the first bed in the series. The second bed then serves as an inherent safety margin removing wastes when the first bed has exhausted its capacity. This operation also provides a built-in safety margin for surges in hydraulic or concentration load.

Reduced operator attention, as compared to a powdered system with mechanical feeders where changing of feed hoppers settings are required.

Based on vendor design information, a rough carbon treatment facility design was calculated. This computation was made based on hydraulic rates and retention time. Two alternatives were sized; a two-parallel train system and a single train system. For both, a train consists of two adsorbers in series. The two-train system consists of four adsorbers, 10' diameter by 10' bed height. The single train system consists of two adsorbers, 12' diameter by 14' bed height. Details of the sizing are given in Appendixes J-3 and J-4. The cost for a two-train system is estimated to be $450,000.

Four carbon treatment options were considered, as listed below. Both the type of carbon treatment (powdered vs granular fixed bed) and the type of final disposal (landfill vs carbon regeneration) were considered. Two regeneration options (contract service vs Pine Bluff Arsenal-owned-and-operated) were studied. The options are as follows:

- Powdered carbon - landfill disposal (existing).
- Granular carbon - contract regeneration.
- Granular carbon - PBA regeneration.
- Granular carbon - landfill disposal.

Costs associated with Option 1 (powdered carbon - landfill disposal) are developed in Appendix J-5. This is the existing treatment employed at the CWTF. Based on the listed assumptions, estimated costs associated with the powdered carbon option are $114,600/year at current rates (250,000 gallons per day) and $271,900/year at mobilization rates (650,000 gallons per day).

Costs associated with Option 2 (granular carbon - contract regeneration) are shown in Appendixes J-6 and J-7. Depending on the degree of loading on the carbon (i.e., how much waste one pound of carbon can adsorb), operating costs are $27,800 to $50,600/year at current rates. Cost savings generated by Option 2 over Option 1 are hence $64,000 to $86,800/year at current rates. Using the estimate for the cost of a two-train granular
carbon fixed bed adsorber system ($450,000), a 5.2 to 7.0 payout is predicted at current rates. At mobilization rates, payout is 2.2 - 3.1 years.

A comparison of Option 3 (granular carbon - PBA regeneration) vs Option 2 (granular carbon - contract regeneration) is given in Appendixes J-8, J-9 and J-10. At the very low volumes of carbon consumption foreseen for PBA, self-owned regeneration is not attractive (payout approximately 20 years).

A comparison of Option 4 (granular carbon - landfill disposal) vs Option 2 (granular carbon - contract regeneration) is given in Appendix J-11. Landfill costs are about 11.1 cents/lb of carbon used plus some nominal cost to transport spent carbon to the landfill site. Initial quotes from one contract regeneration company Calgon, (Appendix J-12), indicates that the cost of regeneration is equivalent to buying virgin carbon (i.e., approximately $1.00/lb for either virgin carbon or regenerated carbon). Hence, the incentive for contract regeneration is just the cost of landfill. If an alternate supplier of granulated carbon could be found at a lower cost than Calgon, then this lower cost could offset the landfill expense. More definite prices from competitive bidders will be required before any final conclusion can be reached. Of course, any reduction in contract regeneration cost will definitely swing the decision in that direction.
4.5 Evaluation of Sludge Removal Systems

The existing PBA waste treatment system (Appendix K-1) uses a settling lagoon prior to discharge of the liquid to the environment. This sludge will eventually require dewatering and permanent disposal (landfill). The study identifies three other types of sludge concentration; i.e., Lamella Settler (Appendix K-3), the Parkson Sludge Dewatering Press (Appendix K-2), and Solid/Sludge dewatering with hydrocyclone (Appendix K-4).

A pilot Sludge Dewatering MMT, 5XX1354, Sludge Volume Reduction and Disposal is being run parallel with the Carbon Treatment or In Plant Reuse MMT, 5XX4231. The Sludge Volume Reduction and Disposal Task will send a sludge-free effluent to the carbon columns and concentrate the sludge with the Lamella Settler and Parkson Sludge Press prior to landfilling.

A third type of solid/sludge dewatering is with a hydrocyclone which handles particles (0.005 to 0.5 mm) (Appendix K-4) effectively and with a minimum of cost. This is the size range between centrifuge and screening processes. This unit will be evaluated and could be used as a pre-treatment to the Lamella Settler to remove the larger particles with the effluent then passing to the Lamella Settler, etc. This unit also has application upstream to remove larger particles prior to adding lime and alum and entry to the Lamella Settler. PBA application could be at the Colored Smoke Production Facility; HC Smoke Facility, Red Phosphorus Production Facility, etc. A pilot hydroclone was purchased for these tests (Appendix K-5) with typical computation for using hydroclones being reported in Appendixes K-6 and K-7. These experiments will be run over the next several years in tandem with all other systems operational as the production program dictates.
4.6 Water Quality Criteria

Toxicity Testing:

The United States Environmental Protection Agency is in the process of changing the emphasis of Water Quality Standards from technology-based effluent limitations to water quality-based limitations, as evidenced by the recent publication of Proposed Water Quality Standards (47FR pp. 49234-49252, October 29, 1982) and the distribution of the DRAFT "Water Quality Standards Handbook" (US EPA Office of Water Regulations and Standards, October, 1982). This shift in regulatory approach for effluent limitations requires a similar shift in the type of data necessary to set the limitations. Toxicity data on effluents will become more important in the future, particularly when dealing with complex mixtures because chemical analyses (Appendix I-1) are not always sufficient for estimating the environmental hazards of a complex effluent. Such analyses do not take into account synergistic or antagonistic effects interacting components may have on organisms. The Federal EPA is beginning to require biomonitoring of effluents, predominantly as part of the second round of NPDES permits (Appendix I-2). In this context, biomonitoring refers to the periodic testing of an effluent, usually on a quarterly basis, for acute toxicity. The point of sampling may vary from end-of-pipe to within the mixing zone of the receiving water. The allowable toxicity is set on a case specific basis.

In anticipation of required period toxicity testing of PBA effluents, the toxicity of effluents after carbon treatment using aquatic organisms was estimated from both acute and chronic toxicity tests using the invertebrate, *Daphnia magna*, and the bluegill sunfish *Lepomis macrochirus* as test organisms. These organisms were chosen because they represent important trophic levels of the receiving streams and are standard test organisms from lists approved by EPA. Untreated PBA effluent (no carbon) is usually toxic to both organisms.

The testing scheme was started with acute screening tests with both organisms to determine if static, acute bioassays were necessary to assess the toxicity. An indication of the chronic toxicity of the effluents was obtained from 21-day reproduction tests with *Daphnia magna*. All test methods were based on US EPA accepted protocols.

Acute Tests

The preliminary toxicity screening test consisted of only controls and 100 percent effluent solutions. Each *Daphnia* test consisted of one 250-ml beaker with 200 ml of effluent and an identical beaker with dechlorinated tap water. The screening tests for the bluegill sunfish followed the same protocol using 19-liter glass test chambers. Each test chamber received ten
test organisms. The invertebrate test duration was 24 hours; the fish test duration was 96 hours.

The results of the screening tests showed no toxicity, thus indicating that full term, acute bioassays on the three effluents were not necessary.

Chronic Tests

Potential chronic toxicity of the effluent to aquatic systems was investigated using *Daphnia magna* reproduction tests. Approximately 40 liters carbon treated (powdered) effluent was shipped to Battelle's aquatic toxicology laboratories. Testing was begun as soon as possible after sample arrival. The effluent from the lagoon had a very dense algal growth and therefore was filtered through a 0.45 um membrane filter before shipment.

The chronic toxicity tests were 21-day static renewal, reproduction tests with *Daphnia magna*. The tests followed ASTM Proposed Standard Practice for Conducting Renewal Life Cycle Toxicity Test with Daphnids (February 6, 1980; Draft 6; Appendix C). Each test consisted of five concentrations of effluent ranging from 100 to 6.24 percent, and controls. The test solutions were renewed three times a week (Monday, Wednesday, and Friday) for the duration of the test. Ten test containers, each containing 200 mls of test solution, were used for each effluent concentration. Seven of the containers at each concentration contained one daphnid each for collection of data on reproduction. Three containers at each concentration contained five daphnids each for collection of data on survival. The number of test containers was doubled for the controls. Juvenile daphnids (less than 24-hours old) were introduced into the test chambers on Day "0". Mortality and reproductive success were assessed three times per week when the test solutions were renewed.

Tests were run at 20 ± 1 degrees C with a light regime of 16 hours light, 8 hours dark. Temperature, dissolved oxygen, pH and specific conductance were recorded at the beginning of the test in all test containers. Each time the test solutions were renewed, the temperature, dissolved oxygen and pH were determined for both the discarded and new solutions in a high, medium and low effluent concentration.

Results

The results of the screening toxicity tests Appendix I-2 indicated that carbon treated effluents were not acutely toxic to either *Daphnia magna* or bluegill sunfish.

Test results showed that reproduction, as evidenced by the number of young per adult, was substantially greater in all test solutions as compared to controls and the reproduction rate was greater in the higher concentrations of effluents. While the
exact cause for this increase in reproduction cannot be fully explained without further testing and analyses, the reproduction of the daphnids in the experimental chambers was probably influenced by food supply. Algae and bacteria in the effluent may have provided an alternate food supply for the Daphnia.

The results of these tests do not indicate that the effluents will cause any significant adverse impact on the receiving streams. This conclusion must be qualified by the fact that these tests were performed once for each effluent. It is recommended that in the future, the toxicity of the effluents be monitored on a fixed time schedule (at minimum, quarterly) to help assess the effects of seasonal changes in receiving stream water flows and quality on the test results, and aid in detecting possible variations in effluent quality.
5.0 **CONCLUSIONS/RECOMMENDATIONS:**

Future carbon column treatment is necessary to meet both current and future EPA requirements for chemical and biological parameters. The trend with EPA guidelines/permits is toward more restrictive standards for both chemicals by name and aquatic toxicity. Currently TOC is a less restrictive parameter which applies to a host of organic chemicals.

Current batch carbon treatment methods need to be replaced by the more effective columns treatment methods to meet future EPA requirements. Batch treatment is also not cost effective and generates a larger volume of sludge to treat prior to landfilling in a RCRA-permitted facility.

A Pilot Plant has been set up in the Central Waste Treatment area of PBA and will be operated over a two-year period to gain technical data relative to PBA's variable and mixed stream waste load. Upon completion of this study, a project will be developed for installation of the equipment necessary to make the required improvements.

Relative to carbon regeneration at PBA this would not appear to be cost effective but rather should be placed in the landfill or regenerated by contract. Since contract and carbon replacement costs appear to be the same, it may be important that the US Army establish a central carbon regeneration facility or go for contract regeneration of the load from the several manufacturing plants in AMCCOM. While landfill may seem to be the way for PBA, (Landfill cost is .11/lb for PBA using MCA 86 cost estimate) this would not be true for high volume carbon generators and those agencies not already having a RCRA landfill programmed. RCRA landfills are costly and require millions to construct. The issue of carbon management for the several US Army Plants needs to be resolved.
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Appendix A

Scope of Work for MMT
Inplant Reuse of Polluted Abated Waters
1. Subject scope of work, dated 4 January 1982, is forwarded for approval/concurrence (Incl 1). The writing of this scope of work was coordinated between representatives of ARRADCOM (DRDAR-LCM-SA) and Pine Bluff Arsenal (SARPB-ETA). The funding level for this work effort will be $160,000.

2. Request approval/concurrence of this scope of work be provided ARRADCOM (DRDAR-LCM-SA) as soon as possible so as not to delay the obligation of FY82 funding to Pine Bluff Arsenal.

3. ARRADCOM POC is Mr. L. Sotsky, DRDAR-LCM-SA, AV880-3544/3007.

FOR THE COMMANDER:

[Signature]

D.J. CASEY
C, Spec Tech Br,
Energetic Sys Proc Div, LCWSL

1 Incl
as
CF:
DRDAR-LCP-T, w/Incl
SCOPE OF WORK

FISCAL YEAR: 82

PROJECT NUMBER: 5824231

PROJECT TITLE: IN-PLANT REUSE OF POLLUTION ABATED WATERS

LABORATORY/DIRECTORATE: LARGE CALIBER WEAPON SYSTEMS LABORATORY
ENERGETIC SYSTEMS PROCESS DIVISION
SPECIAL TECHNOLOGY BRANCH

ESTIMATED FUNDING LEVEL FOR S.O.W.: $160K

TYPE S.O.W.: OGA - PINE BLUFF ARSENAL, PINE BLUFF, AK

RESPONSIBLE LABORATORY/DIRECTORATE ENGINEER: LARRY SOTSKY

OFFICE SYMBOL: DRDAR-LCM-SA

TELEPHONE EXTENSION: AV880-5544/3007

DATE: 4 JANUARY 1982
I. OBJECTIVE:

The objective of this project is to conduct a survey of both the water and chemical utilization in Pine Bluff Arsenal's pollution abatement facility in order that control methods for minimizing flow and chemical utilization as reducing generation of sludge may be identified. As a direct result of this project design criteria will be provided for optimizing the resources utilization of the existing pollution abatement facility. The optimization/modernization will result in reducing both operating costs and sludge generation while maintaining acceptable effluent limits as required by the Clean Water Act, Resource Conservation Recovery Act, and Toxic Substance Control Act.

II. BACKGROUND:

Pine Bluff Arsenal's pollution abatement facility, which treats the industrial waste water to meet National Pollutant Discharge Effluent Standards (NPDES), Toxic Substance Control Act (TSCA), and Resource Conservation Recovery Act (RCRA) regulations, is a heavy consumer of valuable resources: water and chemicals. The industrial waste water is collected from a large production area and delivered to the central waste treatment facility through various lift stations, delivering on demand, which slugs the facility with high concentrations of production chemicals. The flows from the production areas are currently neither monitored nor equalized prior to treatment, thus creating a situation in which chemical feeders must be set at a high rate to treat the periodic slugs and prevent violation of the NPDES permit. The use of excessive treatment chemicals increases the sludge volume that must be treated and/or placed in a toxic landfill in order to comply with RCRA, TSCA, and EPA requirements. These flows should be monitored, minimized, and equalized in order to reduce the consumption of water and chemicals as well as to reduce the amount of sludge generated based on current production requirements.

III. PROCEDURE:

A survey of water and chemical utilization in the pollution abatement facility shall be conducted, identifying control methods to minimize flow, chemical utilization, and reduce sludge generation. An industrial waste water influent equalization basin will provide a uniform waste concentration, minimizing chemical additions and reducing sludge volumes. Evaluation of the use of continuous monitors (TOC, pH, etc.) in an equalization basin, tied to a
microprocessor to provide close control of chemical feeders, could minimize/optimize the chemical and water usage while meeting the environmental regulations (WATER, RCRA, and TSCA).

As part of the FY82 Scope of Work the following will be accomplished:

a. Treatment Chemical Utilization Survey: Central waste treatment influent originates from thirty-seven production buildings, boiler plants, motor pool, and incinerator complex scrubbers, and these produce flow volumes and concentrations with wide variations. This task of the Scope of Work will survey variations in treatment chemical demand and adequacy of equipment response. Areas of operation where quantities of feed chemicals can be reduced will be pinpointed for modification.

b. Identify/Evaluate Optimization Methods: Methods of optimization of chemical feed rates such as pre-equalization of flow and concentration, and automatic controls will be evaluated.

c. Evaluate Use of Continuous Monitors: The use of continuous monitoring and control equipment for significant influent pollutants as a means of optimizing chemical treatment feed rates will be evaluated. Equipment is available for installation that will measure temperature, pH, dissolved oxygen, turbidity, conductivity, ORP (oxidation/reduction potential), and zeta potential of suspended solids. An instrument for the continuous on-line measurement of total organic carbon (TOC) will be purchased and installed. The performance of these instruments will be evaluated, along with their potential to provide optimizing control of chemical feeders. The need for a microprocessor controller unit will also be evaluated.

d. Provide Design Criteria for Optimization System: Design criteria will be established for a system that will provide influent equalization and utilize the continuous monitors/microprocessor controllers to optimize chemical feed rates and yield the best water quality at minimum cost while meeting both EPA and State of Arkansas discharge requirements.

e. Provide Final Technical Report: A final technical report of the project findings and recommendations will be prepared. Typical monitoring data and test results will be included.

IV. REPORTS:

a. A detailed Project Status Report, RCS DRCHT-301, required monthly, will be submitted to ARRA/DCM, DRDAR-LCM-SA, by COB 5th working day of each month. This report will reflect the project's monthly cost/performance in addition to other information requested.
b. On completion of the overall project, a Final Technical Report will be prepared and submitted in final draft form to ARRADCOM, DRDAR-LCM-SA, for review, approval, and final publication. This report is to be prepared in accordance with the format cited in MIL-STD-847A, including all supplements. All data will be expressed in both Metric and US Customary Units of Measurement in accordance with the Metric Practice Guide, ASTM-E-380-74.

V. MILESTONE SCHEDULE:

It is planned to accomplish the Scope of Work described herein within the following milestone schedule:

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<td>Final Technical Report</td>
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VI. PROGRAM APPROVAL:

All technical direction within the bounds of the Scope of Work is the responsibility of ARRADCOM, DRDAR-LCM-SA, and any direction affecting changes to the funding or Scope of Work must be implemented through ARRADCOM, DRDAR-LCM-SA.
Appendix B

Project Financial Documentation
Project Financial Document

Project - Funded MMT .............................................. $160,000

Cost Breakout

Contract - Install Equipment ......................... $29,156

Equipment and Materials
Carbon (9,840 lbs) ........................................ $ 8,840
TOC Analyzer System .......................................... $15,575
Conductivity System .......................................... $ 3,630
pH Controller .................................................. $ 2,920
Hydrocyclone ................................................... $ 5,550
Misc Material .................................................. $ 1,288

SUB TOTAL: .................................................. $37,753

Labor (In House)
Engineering ($39.24/hr X 1,649.06 Hr). $64,709
Shop Labor ...................................................... $28,382

TOTAL ...................................................... $160,000

BALANCE ...................................................... $000,000

1 Estimated 30 Sep 84 Balance. All Funds Committed
Appendix C

Process Flow Chart for Carbon Column Treatment System
PROCESS F

PROCESS WASTE WATER [774 GPM]

CARBON

ALUM

LIME

SUMP

LAMELLA SETTLER

CARBON COLUMN FEED TANK

SLUDGE

TO LAGOON

LEGEND

1. TOC TOTAL ORGANIC CARBON ANALYZE

2. CONDUCTIVITY METER

3. FLOW GAGE

4. FLOW METER

5. SAMPLE PORT

6. PRESSURE GAGE

7. THERMOMETER

8. pH METER

9. HYPASS IN normal PUMP PIP

8/24/84 MD FRAUENTHAL
Appendix D

Process Flow of Installed Pilot Carbon Column Treatment System
D. REMOVAL, AND WASH SYSTEMS

WASTE WATER FEED

SURFACE WASH

BACKWASH

POTABLE WATER
EDUCTOR

CARBON COLUMN B

TREATED WATER

CARBON REMOVAL SYSTEM

POTABLE WATER
EDUCTOR

CARBON COLUMN A

8/24/84 MD FRAUENTHAL
Appendix D-2
Waste Water Desludging Facility
Appendix D-3

Controller for Pilot Carbon Column Treatment Process
Appendix D-4

Pilot Carbon Column Treatment System
Appendix D-5

Pilot Carbon Column Manifold
Appendix D-6

Laboratory Scale Carbon Treatment System
Appendix D-7
Central Waste Treatment Facility Lagoon
Appendix D-8

Lamella Settler Feed Pump
Appendix D-9

Granular Carbon Treatment Facility Prefilters
Appendix D-10

Spent Carbon Receiving Tank
Appendix D-11

Powdered Carbon Feeder System
Appendix E

Process Chemical Assay/Monitoring Equipment
Appendix E-1

Automatic Total Organic Carbon Analyzer
MODEL 700
Automated Total Organic Carbon

O.I. CORPORATION
College Station, Texas
Introducing . . . 

The Model 700 Total Organic Carbon Analyzer—This instrument represents the product of OI’s 15 years of experience in TOC measurement. The 700 is the state of the TOC art—the most accurate and the most highly automated TOC analyzer offered to date. The 700 is capable of analyzing the widest spectrum of sample matrices, yet it is offered at a very competitive price.

The Method is Classic

The oxidation of organics by persulfate at 90-100° C remains the most accurate means of measuring TOC described in the scientific literature. The best evidence that a given methodology is the choice of serious chemists is its literature base. We know of no other TOC method which boasts such an extensive reference library in refereed journals. Some pertinent references are listed on the back. Saline solutions and particulated samples, as well as solutions with hard-to-oxidize materials, do not pose the recovery problem that exists with low temperature-UV methods. Though no method can claim 100% recovery of all compounds in any sample matrix, the persulfate/95° C method exhibits the highest compound recovery efficiencies in the most common sample types (pure waters, waste waters, salt waters) of any presently existing method. Virtually all organic compounds dissolved in water are oxidized with efficiencies of 95% or better.
Model 700 Advantages:

OVER UV/PERSULFATE METHODS:
- Will accurately measure TOC in particulate sample.
- Will accurately measure TOC in saline solutions without sample pre-treatment.

OVER COMBUSTION/IR METHODS:
- Will accurately measure TOC down to 25 ppbC.
- Measures TOC directly, not by difference of total and inorganic carbon.

Model 700 Features:

SEPARATE MEASUREMENT OF:
- Total Organic Carbon (TOC)
- Particulate Organic Carbon (POC)
- Purgeable Organic Carbon (POC)
- On the same sample.

TOTALLY AUTOMATIC:
- Microprocessor controlled timing sequences and temperatures.

CLASSIC METHOD:
- Solid literature base for method establishes unequaled accuracy, especially for salt waters and particulates.
- EPA accepted (EPA 600/4-79-020 Method 415A).

LOOP INJECTION OF SAMPLE:
- Allows easier and more reproducible introduction of sample.
- Also allows unattended online monitoring of process streams.

HUMAN ENGINEERED:
- Simple Operation
- Single Point Calibration
- Digital Display of Concentrations
- rugged, abrasion-proof components and long life
- Operation in Harsh Environments
- Unattended Operation
- Easy Maintenance

OPTIONS FOR EXTENDED CAPABILITIES:
- Purgeable measurements to 10 ppbC.
- Extended sample capability with ampule attachment.

OPTIONS FOR UNATTENDED OPERATION:
- Autosampler
- On-Line Sampling Valve
- Printer
Summary of Method

TOTAL INORGANIC CARBON

Total Inorganic Carbon (TIC) is determined by the measurement of carbon dioxide released by acidification of sample. As pH of the sample is lowered, carbonate and bicarbonate ions are converted to dissolved carbon dioxide. This carbon dioxide is purged from solution, concentrated by trapping, then desorbed and carried into a non-dispersive infrared detector (NDIR) which has been calibrated to directly display the mass of carbon dioxide detected. This mass is equivalent to the mass of TIC in the sample. Concentration of TIC is calculated by dividing this mass by the sample volume.

TOTAL ORGANIC CARBON

Total Organic Carbon (TOC) is determined by the measurement of carbon dioxide released by chemical oxidation of the organic carbon in the sample. After the sample has been acidified and purged of TIC, potassium persulfate (K₂S₂O₈), a strong oxidizer, is added. This oxidant quickly reacts with organic carbon in the sample at 95°C to form carbon dioxide. When the oxidation reaction is complete, the carbon dioxide is purged from the solution, concentrated by trapping, and detected as described for TIC. The resulting mass of carbon dioxide is equivalent to the mass of organic carbon originally in the sample.

Calibration

Calibration of a TOC analyzer has never been simpler. Select calibration mode. A microliter quantity of carbon dioxide gas is injected and the “start button” is pressed. In two minutes, repeat the injection for duplication. Enter the amount of carbon dioxide injected. Calibration is complete. Time required is five (5) minutes with a warmed-up instrument (warm-up takes less than 30 minutes).

Calibration with carbon dioxide provides an absolute calibration of the detector. Carbon dioxide calibration eliminates questions about age or reliability of aqueous standards, or about oxidation efficiency. Also eliminated is the weighing, mixing, or dilution of standard materials. Corrections resulting from organic carbon originally in the water used to prepare TOC standards are not needed.

Alternatively, calibration may be accomplished by injection of an appropriate volume of sodium carbonate, KHP, or other accepted carbon standard solution.
Operation

Step 1:
Refer to Figures A and B. A water sample may be introduced into the instrument either by syringe injection or by means of calibrated sample loop. The sample loop affords greater consistency of injection volume for larger volume samples, whereas the syringe injection port allows injection of microliter quantities of samples of extremely high carbon concentration. Once the sample has been introduced, the START button is pressed and the entire analysis sequence is automatic, beginning with the addition of a metered amount of acid to the sample in the digestion chamber.

Step 2:
Refer to Figure C. After acid has been added, a gas stream purges out any carbon dioxide formed from inorganic carbon in the sample. This carbon dioxide is carried to a molecular sieve trap held at 25°C where it is trapped and concentrated.

Step 3:
Refer to Figure D. When purging and trapping is complete, the trap is placed in line with the infrared detector and rapidly heated to 200°C. A stream of gas desorbs the carbon dioxide from the trap and carries it into the detector. The detector response represents the amount of TIC in the sample. Concentration of TIC is displayed and may be printed.

Step 4:
Refer to Figure E. While the carbon dioxide from TIC is being detected, a metered amount of potassium persulfate is added to the sample, which by now has been heated to 95°C. Gas flow to the digestion vessel has been shut off. The persulfate reacts with organic carbon in the sample to produce carbon dioxide, which accumulates in the digestion vessel.

Step 5:
Refer again to Figures C and D. After a specified reaction time, the digestion vessel is replaced in line with the trap and a gas stream purges out any carbon dioxide produced by the persulfate oxidation. This carbon dioxide is carried to the trap held at 25°C where it is trapped and concentrated.
As before, the trap is then placed in line with the detector and heated. The carbon dioxide is carried into the detector, and the resulting concentration of TOC in the sample is displayed/printed.

Step 6:
Refer to Figure F. While the carbon dioxide from TOC is being detected, gas flow in the digestion chamber is reversed, and the spent sample is carried out of the chamber to drain. The system is ready for a new sample.
Purgeables Option

The automatic measurement of purgeable organic carbon (POC) is offered as an option with the Model 700. This option allows the operator to screen samples for solvent spills and ground water contaminants, and can be used as a possible screen for trihalomethanes since the detection limit is better than 10 ppbC.

Purgeables Method

The POC measurement is accomplished by addition of another trap and furnace to the analysis train (Refer to Figure A). As the sample is being purged of carbon dioxide from TIC, purgeable organic compounds are also removed from the water sample. These compounds are carried by the purge gas stream onto a purgeables trap packed with Tenax GC® where they are trapped and concentrated (other or combination trapping substrates are available upon request). The carbon dioxide from TIC passes through the POC trap and is carried onto the molecular sieve trap. After a pre-determined purge time, the purgeables trap is placed in line with a cupric oxide furnace and detector, and the trap is rapidly heated to 180 degrees C to desorb POC. The desorbed organic compounds are carried into the furnace where they are oxidized to carbon dioxide, the resulting carbon dioxide is detected and the corresponding POC concentration is displayed.
Model 700 TOC — Specifications

1. Operation Mode:
Separate determinations on same sample for Total Inorganic Carbon (TIC), Total Organic Carbon (TOC). Analysis is completely automatic after sample injection. Additional capabilities listed as options.

2. Method - TOC:
Persulfate oxidation at 95°C, purge and trap carbon dioxide, IR detection.

3. Range:
- TOC: 25 ppmC to 10,000 ppmC
- TIC: 10 ppmC to 10,000 ppmC

4. Precision:
Greater of ± 2% or 10 ppmC.

5. Detection:
Linearized non-dispersive infrared (NDIR). Printer optional.

6. Readout:
Digital display of concentration in ppmC.

7. Calibration:
Single point by carbon dioxide injection or prepared standards.

Purgeable Organics Specifications

1. Method:
Purge and trap purgeable organics, oxidize at 600°C to carbon dioxide, IR detection.

2. Range:
10 ppmC to 10,000 ppmC

3. Precision:
Greater of ± 2% or 10 ppmC.

4. Readout:
Digital display of concentration in ppmC.

5. Time of Analysis:
Add two (2) minutes to standard sequence per sample.

6. Dimensions:
Option is internal to base instrument.

Ampule TOC Specifications

1. Method:
Purging and trap carbon dioxide, IR detection.

2. Range:
100 ppmC to 1000 ppmC

3. Precision:
± 1% from 100 ppmC to 1 ppmC and ± 2% for values above 1 ppmC.

4. Readout:
Digital display of concentration in ppmC.

5. Time of Analysis:
Digestion of ampules requires 30 minutes per set. Time of purging cycle is six minutes.

6. Utility Requirements:
Purging and sealing unit power — 115/230 VAC, 50/60 Hz, 400 W

7. Dimensions:
Purging and sealing unit — 41 cm (16 inches) wide x 43 (17 inches) high x 38 cm (15 inches) deep

8. Weight:
Purging and sealing unit — 50 lbs shipping weight

References


Leaderer, B.R. (1978) Summary of the New York Summer Aerosol Study (NYSAS), Air Pollution Control Assoc., 28, Number 4, 321-327.


MODEL 700 PROCESS CAPABILITY

The Model 700 Process Capability (Cat. No. 164-559) consists of a sample loop valve, peristaltic pump, associated tubing, and electronic controls built into the Model 700 to automatically actuate the components at predescribed times in the analysis cycle. With this option, a teflon sampling tube can be placed in a sample bottle and the Model 700 will automatically pump samples through a loop and inject the loop volume into the analysis system. In this manner, the instrument can continually analyze replicates out of the sample bottle until the sample line is placed in the next bottle. Sample loop volume can be changed (0.1 to 10 cc) according to concentration range of samples. The process option also allows the sample loop to be plumbed directly into a source of process water to be analyzed. An alarm relay can be triggered at a preset concentration limit. The analysis sequence can be set to run continually (readout every 10 minutes) or with a delay between analyses of up to 24 hours.
THE MODEL 700 TOTAL ORGANIC CARBON ANALYZER

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EXPANDED CAPABILITIES OPTIONS

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O·F CORPORATION
Graham Rd. at Wellborn Rd. • P. O. Box 2980 • College Station, Texas 77841-2980
Telephone (409) 693-1711—Toll Free 800-445-0016
Cable Address: O.F.C. JSN
TWX No.: 510-892-7944
Appendix E-2

Automated Conductivity Monitor
AUTOMATIC CONDUCTIVITY MONITOR

- Low-cost instrument specifically designed for water treatment systems
- Wide spans check both raw and treated water on same scale.
- Built-in calibration circuit gives easy standardization.
- Automatic temperature compensation on all ranges
- Built-in alarm can initiate control action.
- Panel, pipe or wall mounting — or portable

RECEIVER SPECIFICATIONS

Ranges: 0-10, 0-20, 0-500 and 0-5,000 μS/cm (μmhos/cm)

Meter Scale: 11.4 cm (4.5") long, black on yellow background, with process range as specified. Lower scale is 0-100 with 50 divisions for alarm/control settings. Calibration CHECK position is marked on lower scale.

Limit of Error: Meter: ± 2% of full scale; output (voltage or current) ±1% of full scale

Stability: 1% of full scale per month, noncumulative

Humidity Effect: Negligible at 95% relative humidity over the ambient temperature range

Response Time: One second for 99% of step change

Ambient-Temperature Effect: 0.1% of full scale per degree from –10 to 60 C (14 to 140 F)

Line-Voltage Effect: 0.1% of full scale per volt from 90 to 132 volts (120 V operation) or 198 to 264 volts (240-V operation)

Circuitry: Solid-state feedback-stabilized amplifier for drift-free operation; designed to minimize REL effect

Alarms/Controls: Standard single alarm, for high or low deviation, with setter located on front panel. Set with screw driver or coin to prevent accidental displacement. 0-100 scale with 50 divisions matches lower meter scale for each alarm setting. Red and amber lights indicate conditions above and below set point. Fail-safe alarm selection for "high" or "low" alarm operation.

Contact Rating: 3 A on 120 V a-c or 28 V d-c; 1.5 A on 240 V a-c, inductive load. Hysteresis (dead band): Standard alarm, 2% of full scale, fixed; optional alarm, 1, 3 or 5% of full scale, selectable. Repeatability: 0.25% of full scale. Setting Accuracy: 1% of full scale

Recorder Output: Outputs, with maximum load resistance at ±10% of nominal line voltage, are: 0-16 mA (1300 ohms); 4-20 mA (1000 ohms); 0-20 mA (1000 ohms); 5-25 mA (850 ohms); 0-40 mA (450 ohms); 10-50 mA (330 ohms). 0-1 V and 0-10 V selections also are included.

Temperature Compensation: Automatic compensation is standard.

Power Requirements: 120V (90-132 V), 50/60 Hz, or 240 V (198-264 V) 50/60 Hz, switch-selectable; 12 VA

Electrical Connections: Two 1.90 cm (¾") conduit openings in bottom of case; barrier terminal board inside

Case: Gray, NEMA 12 corrosion-resistant, rain-resistant, dust-proof Noryl plastic case

Dimensions FRONT PANEL: 244 mm (w) x 155 mm (h) x 19 mm (d) (9½" x 6¼" x ¾"). OVERALL DEPTH: 165 mm (6¼"). PANEL CUTOUT: 225 cm (w) x 146 mm (h) (8½") x 3/4".

Weight: 2.5 kg (5.5 lbs)

Hach Company, 1982, 1983, 1984. All rights are reserved

STANDARD CELL

STANDARD CELL IN PVC HOUSING
CONDUCTIVITY CELLS

Features universal mounting for insertion, immersion or flow. Designed to measure effluent of distillation equipment and ion exchangers of the cationic, anionic and mixed-bed type; also being used for measuring effluents of micro-electronic washing and plating rinse tanks. Features PVDC construction for high corrosion resistance.

CONDUCTIVITY CELL SPECIFICATIONS

Function: Designed for installation by insertion in pipe or immersion in a tank, or for installation in a process stream or by-pass line.

Cell Constant: As specified*

Electrode Material: Gold-plated nickel

Maximum Pressure Limit: 2170 kPa (300 psig) at 25°C; to 1632 kPa (225 psig) at 50°C; 790 kPa (100 psig) at 75°C; 273 kPa (25 psig) at 100°C

Maximum Temperature Limit: 100°C for all ranges

Insertion Depth: 152 mm (6") below threads; 25 mm (1") NPT** Male for Schedule 40 Pipe, 32 mm (1.25") NPT Male for Schedule 80 Pipe. Use reducing bushing with Schedule 80 Pipe.

Immersion Depth: up to 1.8 m (6') with standard 2.1 m (7') cable

Flow: Mount directly in cell housing inlet; 19 mm (0.75") NPT Male; outlet, 19 mm (0.75") NPT Female

CONDUCTIVITY RECEIVER MOUNTING STYLES

Every Receiver comes complete with hardware for panel, wall, or pipe mounting.

Pipe Mounting — Suspended below horizontal pipe, or attached to vertical pipe on right side, left side, or rear.

Panel Mounting — Same two angle brackets mount the monitor in 225 x 146 mm (8.875 x 5.75") panel cutout.

Wall Mounting — Reverse the angle brackets and mount the monitor on any flat surface.

HOW TO ORDER

Conductivity Receiver, (specify range) 19456-00
Conductivity Cell 19457-00
Junction Box to extend cable 19458-00
Extra Cable, priced per foot 7425-07

In the United States, call toll-free 800-525-5940. Colorado residents call toll-free 800-227-4224.
Appendix E-3

Gas Chromatography Method for Hexachloroethane
GC Method for Hexachloroethane in Water Using a Hexane Extraction

I. Extraction Procedure

1. Put 10ml of water into a culture tube with cap.
2. Add 1ml of hexane.
3. Vortex for five minutes.
4. Centrifuge for ten minutes at 2,000 RPM.
5. Pipette off the hexane and put into an evaporating tube.
6. Using nitrogen or air, evaporate hexane to 0.1ml.

Extraction technique better than 90% efficient.

II. G. C. Parameters

A. Column

1. 10 ft, 1/4" OD, 2mm ID, glass
2. 1.50% SP2250. and 1.95% SP2Y01 on 100/120 mesh Supelcoport GC column packing material
   or
   1.50% OV17 and 1.95% OV210 on 100/1230 mesh
3. Ensure column has been silylated.

B. Integrator Parameters

1. Zero = 15
2. Attn 2 = -2
3. Chl Sp = 0.5
4. Pk Wd = 0.16
5. Thrsh = -2
6. Ar Rej = 0

C. Instrument Parameters

1. Isothermal Operation
   a. Initial Oven Temperature = 75°C
   b. Initial Hold Time = 8 min
2. Right Detector, EC
a. SAT Current \( = 1.5 \times 10^9 \text{A} \)
b. Temperature \( = 350^\circ \text{C} \)
c. Carrier \( = \text{Ar/CH}_4 \)
d. Mode \( = \text{Linear} \)
e. Filter \( = \text{On} \)
f. Purge \( = 20 \text{ ml/min Ar/CH}_4 \)

3. Right Injector Temperature \( = 200^\circ \text{C} \)

4. Oven Maximum Temperature \( = 250^\circ \text{C} \)

5. Total Run Time \( = 8 \text{ minutes} \)

6. Input \( = 1 \)

7. Polarity \( = + \)

8. Filter \( = \text{M} \)

D. Gas flow parameters

60 ml/min Argon with 5% methane

E. Standards

Range from 1 ppb to 100 ppb hexachloroethane in hexane
Appendix F

Technical Data Sheets on Activated Carbon
Appendix F-1

Technical Data Sheet
Calgon Filtrasorb 300 and 400 Activated Carbon Product
description

Filtrasorb 300 and 400 are two grades of granular activated carbon made by Calgon Corporation to remove impurities from municipal or industrial waste water. Both products are high density carbons, with large surface areas. Their pore structure and mesh size have been carefully controlled for the adsorption of both high and low molecular weight impurities from waste water. Filtrasorb wets readily and does not float. Its high density allows convenient backwashing under conventional flow rate conditions.

purposes

Filtrasorb 300 and 400 are designed for the purification of waste water by removing organic contaminants whether measured as BOD, COD, TOC, color, odor, optical density, or by other analytical techniques. Its dual role of filtration/adsorption is a concurrent function that filters out suspended solids and adsorbs other dissolved organic compounds.

Granular carbon treatment has built-in flexibility since the waste water can be either upgraded partially to meet effluent treatment and receiving stream standards or upgraded completely for direct re-use in high quality water applications. In addition, valuable by-products sometimes can be adsorbed in granular carbon beds and then recovered from the carbon by a chemical regeneration process.

For unit processes dealing with ion exchange or inorganic removal, granular carbon is used to protect the resins and/or membranes which would become fouled by the organic impurities in the waste.

The following are major areas of water treatment where a process utilizing Filtrasorb can purify waste waters beyond levels normally obtainable by conventional biological secondary waste treatment systems:

**Calgon Polysorb Process**—This is a physical-chemical waste water treatment process that can produce secondary or tertiary quality water. The process clarifies raw sewage in the primary plant by utilizing water-soluble polymers to maximize removal of suspended solids. The clarified sewage is then percolated through Filtrasorb beds which remove the remaining suspended solids and adsorb the dissolved organics.

**Advanced Waste Water Treatment**—A process utilizing Filtrasorb can be incorporated as a final polishing step. It can be used to complement industrial and municipal waste water treatment systems whose sedimentation, clarification, and bacteriological techniques are inadequate for meeting higher standards of effluent quality.

**Industrial Process and Waste Water Treatment**—Filtrasorb can be incorporated into manufacturing process systems to remove particular types of pollution. It can be used in by-product recovery operations and in industrial waste treatment programs for the removal of phenols, colors, insecticides, TNT, detergents, polylols, dyes and other wastes.

* Polysorb Process is a Service Mark of Calgon Corporation.

advantages

A Filtrasorb carbon waste treatment system offers the following superior performance factors:

* **Effectiveness**—The process is capable of reducing organic contamination considerably below any other proven unit process and accomplishes this at reasonable treatment costs.
Trouble-Free Operation—The Filtrasorb system does not create disposal or pollution problems. Impurities are separated from the water and concentrated on the carbon surface thus permitting easy removal by a thermal reactivation process.

Reactivation—Filt rasorb is manufactured specifically for thermal reactivation and re-use. Numerous reactivation installations have demonstrated the feasibility and economy of reactivating granular carbon. The carbon is easily transported as a water slurry between the filters and the furnace area. No additional manpower or labor skills are required to operate a reactivation facility. The process is automated to run with minimal attention. (Bulletin No. 20-5a describes the reactivation sequence in greater detail.)

Reserve Capacity—The adsorption capacity of carbon can effectively process sudden fluctuations of impurity concentrations due to spills, peak loads, or pre-treatment upsets.

Economy—Reactivation allows re-use of the carbon.

Flexibility—Quality of the effluent can be selected to meet re-use or discharge requirements.

Reliability—Granular carbon adsorption and reactivation of the exhausted carbon for re-use are proven processes.

physical properties

<table>
<thead>
<tr>
<th>FILTRASORB</th>
<th>FILTRASORB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total surface area (N₂, BET method) m²/g</td>
<td>300</td>
</tr>
<tr>
<td>Bulk density, lbs/ft.³</td>
<td>26</td>
</tr>
<tr>
<td>Particle density wetted in water g/cc</td>
<td>1.3-1.4</td>
</tr>
<tr>
<td>Pore volume cc/g</td>
<td>0.85</td>
</tr>
<tr>
<td>Effective size mm</td>
<td>0.8-0.9</td>
</tr>
<tr>
<td>Uniformity coefficient</td>
<td>1.9 or less</td>
</tr>
</tbody>
</table>

*To be used to calculate volume requirements.

Specifications

<table>
<thead>
<tr>
<th>FILTRASORB 300</th>
<th>FILTRASORB 400</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specification Value</td>
<td>Typical Analysis</td>
</tr>
<tr>
<td>U.S. Standard Series</td>
<td></td>
</tr>
<tr>
<td>Sieve Size</td>
<td></td>
</tr>
<tr>
<td>Larger than No. 8</td>
<td>Max. 8%</td>
</tr>
<tr>
<td>Smaller than No. 30</td>
<td>Max. 5%</td>
</tr>
<tr>
<td>Larger than No. 12</td>
<td>—</td>
</tr>
<tr>
<td>Smaller than No. 40</td>
<td>—</td>
</tr>
<tr>
<td>Mean Particle Dia. mm</td>
<td>1.5-1.7</td>
</tr>
<tr>
<td>Iodine Number, min.</td>
<td>950</td>
</tr>
<tr>
<td>Abrasion Number, min.</td>
<td>70</td>
</tr>
<tr>
<td>Ash</td>
<td>Max.</td>
</tr>
<tr>
<td>Moisture</td>
<td>Max.</td>
</tr>
</tbody>
</table>

Packaging

Filtrasorb is packaged in four-ply polyethylene reinforced kraft bags, 60 lbs. net, 61 lbs. gross. Available on trailer-truck pallets, 42" x 54", maximum 28 bags per pallet—16 pallets maximum number for a standard 40 ft. trailer; or on rail car pallets, 48" x 48", maximum 42 bags per pallet—24 pallets maximum number per standard 50 ft. rail car. Bulk shipments by truck 30,000 lbs. minimum and by rail 50,000 lbs. minimum. Less than ton quantities are packaged in protective shipping cartons—tare weight of carton—2 lbs. Shipping point: Catlettsburg, Kentucky.

For further information write to Filtrasorb Department, Water Management Division, Calgon Corporation, P.O. Box 134, Pittsburgh, Pa. 15230.
Granular Activated Carbon Products
Remove Dissolved Organic Chemicals from Industrial and Municipal Wastewaters Economically and Reliably

Filtrasorb 300 and 400 are two grades of granular activated carbon manufactured by Calgon for removal of organic pollutants from industrial and municipal wastewaters. These carbons are manufactured from select grades of bituminous coal to produce a high-density, high surface area, durable granular product capable of withstanding the abrasion and dynamics associated with repeated reactivation, hydraulic transport, backwashing and mechanical handling.

purposes
Filtrasorb granular activated carbons can perform a dual role of adsorbing dissolved organic contaminants and filtering suspended solids. Filtrasorb is effective in removing from wastewaters many dissolved toxic organic chemicals listed by the EPA as priority pollutants and hazardous substances. These include benzene, phenols, dieldrin, toluene, DDT, polychlorinated biphenyls and many others. It is also effective in removing refractory organic chemicals not responsive to biological treatment.

Industrial Wastewater Treatment applications can include the following:
- Applying point source treatment to remove chemicals
- Pre-treatment to biological waste treatment systems
- Product recovery from wastewater

A "point source" Filtrasorb adsorption system at a chemical plant cleans up toxic organics before discharging effluent. Wastes of this type can upset the effectiveness of biological-type treatment operations.

© Calgon Corporation, 1978
Recycling wastewater
Polishing effluent from biological waste treatment systems
Providing total wastewater treatment

Municipal Wastewater Treatment applications can include the following:

- **Physical/chemical treatment** — Filtrasorb carbon is used in conjunction with water-soluble polymers. After solids settling with polymers, the effluent from the primary basin is passed through beds of Filtrasorb to filter out the remaining solids and to remove dissolved organics by adsorption.

- **Advanced wastewater treatment** — Filtrasorb can be utilized:
  a) to polish effluent from a biological wastewater treatment system in order to remove dissolved organic chemicals which were refractory to biological treatment.
  b) to recycle the treated water for replacement of groundwater or for other suitable recycling applications.

advantages and benefits

- **Provides ultimate disposal of pollutants** — When Filtrasorb carbon is saturated with impurities, the process of thermal reactivation destroys the impurities at very high temperatures in the reactivation furnace. This includes destruction of refractory and toxic chemicals which would otherwise be hard to destroy.

- **Economical to use** — Because of its high surface area and abrasion resistance, Filtrasorb can be reactivated repeatedly and returned to service to provide maximum economy.

- **Effective in treating a wide range of impurities** — Filtrasorb carbon removes hundreds of specific organic compounds from wastewaters, including many on the EPA lists of priority pollutants and hazardous substances.

  These carbons are produced with an exceptionally high internal surface area of optimum pore size for adsorption of both high and low molecular weight pollutants.

- **High surface area** — Because of its numerous pores in a wide range of sizes, Filtrasorb provides a very high capacity and efficiency for removing dissolved organics. Total surface area measures approximately 950 to 1050 square meters per gram (N₂, BET procedure).

- **Reliability** — Systems using Filtrasorb carbons accommodate changes in flow rates and increases in concentration of pollutants whether caused by spills, peak loads, pre-treatment upsets or other variations in the wastewater effluent.

- **Suitable for backwash operations** — Filtrasorb carbons are of high density, wet readily, and do not float, thus minimizing loss during backwash operations.

  For the purpose of estimating the volume of Filtrasorb in a system utilizing backwash procedures, Filtrasorb 300 has a backwashed and settled bulk density of 66-7 lbs. per cubic foot, and Filtrasorb 400 has a backwashed and settled bulk density of 25-26 lbs. per cubic foot.

recovery

Numerous installations have demonstrated the feasibility and economy of thermal reactivation.

Exhausted granular carbon can be reactivated on your plant site in a high-temperature furnace, or it can be done by Calgon. Because of the large volume of industrial wastes treated in this municipal plant, the use of Filtrasorb carbon provides greater reliability than a biological treatment system.
Corporation under a service agreement. In either case, a Calgon adsorption system specialist will assist you in comparing the economics and procedures of your reactivation options.

**Specifications**

<table>
<thead>
<tr>
<th></th>
<th>Filtrasorb 300</th>
<th>Filtrasorb 400</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iodine Number (min.)</td>
<td>900</td>
<td>1000</td>
</tr>
<tr>
<td>Abrasion Number (min.)</td>
<td>75</td>
<td>75</td>
</tr>
<tr>
<td>Moisture (max.)</td>
<td>2.0%</td>
<td>2.0%</td>
</tr>
<tr>
<td>Mean Particle Diameter (mm)</td>
<td>1.5-1.7</td>
<td>0.9-1.1</td>
</tr>
<tr>
<td>Effective Size (mm)</td>
<td>0.8-0.9</td>
<td>0.55-0.65</td>
</tr>
<tr>
<td>Water Soluble Ash (max.)</td>
<td>0.5%</td>
<td>0.5%</td>
</tr>
</tbody>
</table>

U. S. Standard Sieve Size:
- Larger than No. 8 (max.) 15%
- Smaller than No. 30 (max.) 4%
- Larger than No. 12 (max.) — 5%
- Smaller than No. 40 (max.) — 4%

**Packaging**

Filtrasorb can be supplied in four-ply polyethylene reinforced kraft bags 25 kg. (55.1 lbs.) net, bulk packs, or bulk shipped by rail car or truck. Shipping point: Catlettsburg, Kentucky.

**Caution**

Wet activated carbon preferentially removes oxygen from air. In closed or partially closed containers and vessels, oxygen depletion may reach hazardous levels. If workers are to enter a vessel containing carbon, appropriate sampling and work procedures for potentially low-oxygen spaces should be followed, including all applicable Federal and State requirements.

**Downflow Pressure Drop**

Through a Backwashed Segregated Bed of Filtrasorb 300 and Filtrasorb 400

![Downflow Pressure Drop Diagram](image)
PERCENT BED EXPANSION OF A BACKWASHED-SEGREGATED BED OF FILTRASORB 300

For other MPDs determine required flow as follows:

\[
\text{GPM/FT}^2_{\text{New}} = \frac{\text{GPM/FT}^2_{\text{Graph}} \times \text{MPD}}{1.6}
\]

Percent Bed Expansion of a Backwashed-Segregated Bed of Filtrasorb 300 - Mean Particle Diameter (MPD) = 1.6

WATER TEMPERATURES

PERCENT BED EXPANSION OF A BACKWASHED-SEGREGATED BED OF FILTRASORB 400

For other MPDs determine required flow as follows:

\[
\text{GPM/FT}^2_{\text{New}} = \frac{\text{GPM/FT}^2_{\text{Graph}} \times \text{MPD}}{1.6}
\]

Percent Bed Expansion of a Backwashed-Segregated Bed of Filtrasorb 400 - Mean Particle Diameter (MPD) = 1.0

For information regarding incidents involving human and environmental exposure, call (412) 777-8900 and request to speak to Regulatory and Trade Affairs.

For additional information, contact the Activated Carbon Division, Calgon Corporation, Calgon Center, Box 1346, Pittsburgh, PA 15230

SUBSIDIARY OF MERCK & CO., INC.
Appendix F-2

Technical Data Sheet
Nuclor Granular Activated Carbons
Nuchar WV-L

Description and Typical Applications

*Nuchar WV-L* is a hard, regenerable, granular activated carbon produced from selected grades of bituminous coal. The broad spectrum of internal pore sizes insures the adsorption of a wide variety of molecular weight organics. Applications where *Nuchar WV-L* is particularly effective include the removal of organics from potable water and the treatment of municipal and industrial wastewaters.

The density and particle size distribution make *Nuchar WV-L* well-suited for packed-bed adsorption applications. Its high resistance to abrasion results in minimum losses during thermal regeneration and mechanical handling. It can be employed in either fixed or moving bed processes.

*Nuchar WV-L* 3x30 can normally be regenerated by thermal reactivation. In some cases it can be regenerated by chemical treatment or steaming in place.

Specifications

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Abrasion Number (Ro-Tap)</td>
<td>70 min</td>
</tr>
<tr>
<td>Iodine Number (mg/g)</td>
<td>950 min</td>
</tr>
<tr>
<td>Moisture, as packed (%)</td>
<td>2.0 max</td>
</tr>
<tr>
<td>Water Solubles (%)</td>
<td>1.0 max</td>
</tr>
<tr>
<td>Particle Size (U.S. Sieve Series)</td>
<td>8 x 30</td>
</tr>
<tr>
<td>Oversize (%)</td>
<td>8.0 max</td>
</tr>
<tr>
<td>Undersize (%)</td>
<td>5.0 max</td>
</tr>
<tr>
<td>Uniformity Coefficient</td>
<td>1.8 max</td>
</tr>
</tbody>
</table>

Typical Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apparent Density (lb/cu ft)</td>
<td>29-31</td>
</tr>
<tr>
<td>Apparent Density (kg/m³)</td>
<td>465-497</td>
</tr>
<tr>
<td>Bed Density, Backwashed and Settled (lb/cu ft)</td>
<td>25-27</td>
</tr>
<tr>
<td>Bed Density, Backwashed and Settled (kg/m³)</td>
<td>401-433</td>
</tr>
<tr>
<td>Effective Size (mm)</td>
<td>0.85-1.05</td>
</tr>
<tr>
<td>Mean Particle Diameter (mm)</td>
<td>1.40-1.70</td>
</tr>
<tr>
<td>Particle Density, Wetted in Water (g/cm³)</td>
<td>1.35-1.45</td>
</tr>
<tr>
<td>Surface Area (Nitrogen BET Method) (m²/g)</td>
<td>1000 min</td>
</tr>
<tr>
<td>Voids in Packed Bed (%)</td>
<td></td>
</tr>
<tr>
<td>Backwashed and Settled</td>
<td>45-55</td>
</tr>
<tr>
<td>Poured Dry Fill</td>
<td>40-45</td>
</tr>
</tbody>
</table>

*Specifications and typical property data using Westvaco procedures.

†Typical properties are for general information and are not to be construed as purchase specifications.

The information contained herein is based on our best knowledge and belief.

It is given gratuitously and without warranty expressed or implied.

nor is there any intention to infringe on any existing or pending patents.

Supersedes all previous Nuchar WV-L Data Bulletins.
Packaging

*Nuchar WV-L* is shipped in bulk trucks, bulk boxes, or in multiwall paper bags in telescoping, corrugated boxes. The bulk boxes and bags-in-a-box are shipped on nonreturnable, wooden pallets with four-way double entry.

**Pressure Drop Characteristics**

The pressure drop per foot of backwashed and settled bed depth is presented in Figure 1 for various superficial velocities with water at 25°C. For other conditions the pressure drop may be calculated from the equation:

\[ h_f = \frac{K \nu V_s L}{g} \left( \frac{1}{d_{50}} \right)^2 \]

where:

- \( h_f \) = head loss due to friction (ft of H2O)
- \( K \) = a constant equal to 550
- \( \nu \) = kinematic viscosity (sq ft/sec)
- \( V_s \) = superficial velocity (ft/sec)
- \( L \) = backwashed and settled bed depth (ft)
- \( d_{50} \) = mean particle diameter (ft)
- \( g \) = acceleration due to gravity (32.2 ft/sec²)

**Backwash Characteristics**

The bed expansion achieved at various backwash rates is shown in Figure 2 for water at 25°C (77°F). The expansion is expressed as a percentage of the backwashed and settled bed depth.

**CAUTION:** Never enter tanks or other confined areas containing wet, activated carbon. Wet, activated carbon will adsorb oxygen and asphyxiation may result.

**CAS Registry 87440-44-0**

**Westvaco**

Chemical Division
Carbon Department
Covington, Virginia 24426
(703) 962-1121
Appendix G

Basic Concepts of Adsorption on Activated Carbon
Appendix G-1

Basic Concepts of Adsorption on Activated Carbon

Calgon Corporation
BASIC CONCEPTS OF ADSORPTION ON ACTIVATED CARBON

Filtrasorb Department
Water Management Division
Calgon Corporation
P. O. Box 1346
Pittsburgh, Pa. 15230

CALGON CORPORATION
SUBSIDIARY OF MERCK & CO., INC.
MICROCOPY RESOLUTION TEST CHART
We have attempted in the following discussion to outline in a simple manner the answers to two questions we are frequently asked:

"WHAT IS ADSORPTION?"

"WHAT IS ACTIVATED CARBON?"

These questions do not have simple answers because of the lack of any single unifying principle which can explain all adsorption phenomena.

Through the years, by working with Activated Carbon both in the research laboratory and in the field, we have evolved our own interpretation of fundamental concepts which we present herein. Some of what we say must be presented without rigorous proof; but the sum-total, we feel, is a logical, self-consistent theory which has explained a host of experimental observations and has guided us to the successful solution of many practical problems.

Though carbons produced by other manufacturers have been mentioned by name, these are included only as examples to show the range of properties found. No implications are made about the relative worth of these carbons as each has its particular merits in respect to specific applications.
Section 1
SURFACE PROPERTIES OF ACTIVATED CARBON

The two principal physical features of activated carbon, upon which its properties of commercial value depend, are surface and pore structure. The role that surface plays can only be understood in relation to the phenomenon of adsorption.

A. THE NATURE OF ADSORPTION

Adsorption is usually explained in terms of the surface tension (or energy per unit area) of the solid. Molecules in the interior of any solid are subjected to equal forces in all directions, whereas molecules in the surface are subjected to unbalanced forces. The resulting inward forces can only be satisfied if other molecules, usually gaseous or liquid, become attached to the surface. The attractive forces are the same as those responsible for surface tension and condensation in liquids. They are relatively weak and are called Van der Waal's forces. When such forces are involved, the adsorption is termed "physical"; and the adsorbed molecules are easily released from the surface, i.e., "desorbed". In contrast to this, chemisorption is the result of chemical interaction with surface molecules. Stronger forces are involved, and the process is irreversible; that is, the molecules originally adsorbed are recovered in the form of compounds containing atoms of the adsorbent. Both physical and chemisorption are included in the general term "adsorption".

There is at present no known method of measuring the surface tension of a solid directly. However, the total surface energy is equal to the product of the surface energy per unit area and the total surface area. For this reason, high surface area is the prime consideration in adsorption; and activated carbon usually have surface areas in the order of 500-1400 square meters per gram.

B. MEASUREMENT OF SURFACE AREA OF SOLIDS

1. The surface area of carbon may be determined by the Brunauer, Emmett & Teller (BET method). The volume of nitrogen gas adsorbed at liquid nitrogen temperature (~195°C.) is measured at various pressures. A plot of volume adsorbed versus pressure at constant temperature is called an adsorption isotherm. By application of the BET equation to the isotherm, it is possible to calculate the volume and, hence, the surface area of a layer of nitrogen one molecule thick. The surface areas, usually expressed in square meters per gram, of some commercial activated carbons are listed below.

<table>
<thead>
<tr>
<th>TABLE 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>SURFACE AREAS OF ACTIVATED CARBON (M²/g.)</td>
</tr>
<tr>
<td>PCC SGL</td>
</tr>
<tr>
<td>PCC BPL</td>
</tr>
<tr>
<td>PCC RB</td>
</tr>
<tr>
<td>PCC GW</td>
</tr>
<tr>
<td>Columbia CXA/SXA</td>
</tr>
<tr>
<td>Columbia AC</td>
</tr>
<tr>
<td>Columbia G</td>
</tr>
<tr>
<td>Darco 51</td>
</tr>
<tr>
<td>Darco 60</td>
</tr>
<tr>
<td>Darco KB</td>
</tr>
<tr>
<td>Hydro Darco</td>
</tr>
<tr>
<td>Nuchar Aqua</td>
</tr>
<tr>
<td>Nuchar C</td>
</tr>
<tr>
<td>Nuchar (various)</td>
</tr>
<tr>
<td>Norit (various)</td>
</tr>
</tbody>
</table>

2. The Harkins-Jura gas method may also be employed. In the case of activated carbon, it has been found that adsorption from solution of small molecules such as iodine gives a good approximation of the surface area. The following methods are good only for non-porous solids.

- Sedimentation methods
- Permeability methods
- Light and electron microscopy
- X-ray low-angle scattering

C. THE CHEMICAL NATURE OF THE SURFACE

Adsorption may be influenced by the nature of the surface and the adsorbate. In the case of solution, it is often said that "like" dissolves "like"; and we may also apply this principle to adsorption and say that "like" adsorbs "like", polar surfaces preferring polar adsorbates and so on. Polar compounds are those which may exist as positive or negative ions or are influenced by an electric field. Most inorganic compounds fall into this category, as well as certain unsymmetrical organic molecules.

A pure carbon surface is considered to be non-polar; but in actual practice, some carbon-oxygen complexes are usually present which render the surface slightly polar. Since there are no satisfactory methods of determining quantitatively the polar character of a surface, the above statement of principle is relative. In general, activated carbon...
is a poor adsorbent of inorganic electrolytes. Due to its higher surface area per gram, activated carbon will adsorb aromatic and unsaturated aliphatic compounds to a greater degree than silica gel which has a polar surface. However, silica gel exhibits pronounced selectivity for unsaturated, in preference to saturated compounds, which is not shown by carbon. This is probably due to the polar nature of the C=C double bond.

Certain carbons, especially of vegetable origin, have an alkaline ash and an alkaline surface which has definite hydrophilic (water adsorbing) properties as will be shown later in its effect on water isotherms. Polar surfaces of this kind may influence the adsorption of polar bodies; e.g., unsaturated organic compounds, dyes and color bodies, but as yet, there is insufficient data to correlate such observations.

It should be emphasized that for activated carbon in general, the chemical nature of the surface is of minor significance and should be considered secondary in relation to the major factor, magnitude of surface area. Though many investigators have looked to the chemical nature of the surface (surface complex, activated sites) for the explanation of anomalies of adsorption, a more satisfying and logical explanation can be found in a "molecular screening" concept to be discussed in a later section.

*Lewis, Gilliland, Chertow and Cadogan, Ind. Eng. Chem. 42, 1319 (1950).*

Section II
PORE STRUCTURE PROPERTIES OF ACTIVATED CARBON

A. CAPILLARIES IN CARBON

The submicroscopic structure of activated carbon is not known with certainty, but it is probably composed of amorphous particles randomly distributed to give a complex network of irregularly-shaped and partly interconnected passages between the particles. For the purpose of arriving at a quantitative concept of size, however, the assumption may be made that these passages or pores are cylindrical in shape.

Evidence for the existence of pores in activated carbon is to be found in the following facts.

1. Large quantities of vapor or liquid are adsorbed (e.g., 60-70 g. CCl4 per 100 g. carbon) without appreciable change in the size of the granule.

2. The difference between the granule volume and the actual volume of the carbon as measured by helium displacement is usually considerable.

3. It is possible to force mercury under pressure into evacuated carbon granules.

4. When the desorption isotherm does not coincide with the adsorption isotherm, it is regarded as evidence that fine capillaries are present. This phenomenon, known as hysteresis, usually occurs when condensible vapors are desorbed from activated carbon, as shown in Figure 1.

5. The large surface area of activated carbon compared to the small geometric area of the particles or granules requires the existence of considerable internal surface which can only be provided by small capillaries.

A variety of research techniques has shown that the pores in activated carbon are divided into two distinct classes in respect to size. The more important pore system, since it contributes nearly all the surface available for adsorptive purposes, is the smaller or "micropore" system. This system is largely the product of the activation process. The larger capillaries, called "macropores" are more dependent on the raw material and preliminary manufacturing process, e.g., grinding and agglomeration of raw material. These pores contribute very little to the surface area but serve as avenues of entrance to the interior of the gross carbon particles. These two systems are discussed separately below.

B. MICROPOR STRUCTURE

Micropores may be arbitrarily defined as pores whose diameters range from 10 to 1000 Å. The most satisfactory method of characterizing micro-
pore size in activated carbons is that described by Juhola & Wiig, using the water desorption isotherm. As already mentioned, most activated carbons possess a surface which water does not wet readily. Water molecules, prefer, to associate, with each other, rather than with carbon. Consequently, at low vapor pressure very little water vapor is taken up by activated carbon. At higher vapor pressures water is readily adsorbed, but this is believed to occur by condensation in the pores and not by adsorption on the surface. On desorbing, hysteresis is noted. It is believed that the desorption isotherm represents the equilibrium involved in emptying pores, and that application of the Kelvin Equation to the desorption branch will permit a quantitative evaluation of pore size. This appears to be substantiated by the fact that surface areas calculated from the resulting pore size distribution are in essential accord with those measured by the BET method.

1. Determination of Micropore Structure by the Kelvin Equation

The Kelvin Equation is derived from thermodynamic considerations of the free energy changes involved in the process of transferring liquid from unadsorbed phase to adsorbed phase in fine capillaries. The equation is as follows:

$$D = \frac{4 \sigma V \cos \theta}{RT \ln (P/P_0)}$$

Where

- $D$ = pore diameter
- $\sigma$ = surface tension of adsorbed water
- $V$ = molar volume of adsorbed water
- $\theta$ = carbon-water contact angle
- $R$ = gas constant per mole
- $T$ = absolute temperature
- $P$ = vapor pressure of water
- $P_0$ = saturation vapor pressure of water

If $\sigma$, $V$, $\cos \theta$, $R$ and $T$ are considered constant, the equation reduces to the simple form

$$D = \frac{1}{k \ln (P/P_0)} \quad \text{or} \quad \frac{1}{D} = k \ln (P/P_0)$$

The reciprocal of the pore diameter is proportional to the natural logarithm of the relative vapor pressure $(P/P_0)$. Pore diameter values may be superimposed on the $P/P_0$ scale of the water isotherm to give a reasonable indication of the pore size distribution in the micropore range. Above $P/P_0 = 0.99$, $\ln (P/P_0)$ and $1/D$ approach 0. $D$ rapidly approaches infinite size and accuracy in pore diameter measurement becomes poor. Consequently, reliable pore diameter values greater than 1000 Å are difficult to obtain by means of the Kelvin Equation.

2. Interpretation of the Water Desorption Isotherm by Inspection

Activated carbons are readily differentiated by water isotherms. The relationships between structure and adsorptive tests will be developed in Section III. However, the manner in which Iodine and Molasses Numbers are affected by the shape of the water isotherm may be illustrated by the following diagrams. The left-hand, middle and right-hand portions of the isotherms will be referred to as regions 1, 2 and 3 respectively.

Let us assume that the carbon in Figure 2 has an iodine number of 1000 and molasses number of 150. If the steep part in region 2 is raised as in Figure 3, the result will be an increase in iodine number, say, to 1200 with no change in the molasses number. If, however, the slope in region 3 only is increased as in Figure 4, there will be little change in iodine number.

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*Juhola & Wiig, J. Am. Chem. Soc. 71, 2069 (1949).*
but appreciable increase in molasses number, say, to 200. Slight movement of the steep part in region 2 to the right as in Figure 5 should result in a decrease in iodine number with little change in molasses number; but if this is carried further, a point will be reached where a very large increase in molasses number will occur due to enlargement of pores beyond the minimum diameter for molecules. Fortunately, the occurrence of molasses color bodies. Figure 2 would correspond to BP carbon, Figure 3 to Columbia nutshell and Figure 4 to SG. In the case of RB and decolorizing carbons with high molasses numbers, the slope in region 3 is exceptionally steep. As a general rule, the steeper the isotherm in region 3, the greater will be the decolorizing power of the carbon.

Due to the hydrophobic nature of most carbon surfaces, the isotherm in region 1 is practically horizontal. However, a hydrophilic surface, caused perhaps by ionized or polar groups is recognizable by the fact that the isotherm has an appreciable slope at low vapor pressures and approaches the origin at an angle, as illustrated by the isotherm of Darco carbon (Figure 6). When this occurs, the lower part of the isotherm (region 1) cannot be used to evaluate realistic pore diameters. Fortunately, this hydropilic tendency can usually be destroyed for experimental purposes by acid or water extraction and calcination of the carbon.

C. MACROPORE STRUCTURE

Since pore diameters greater than 1000 Å cannot be calculated with accuracy from a water isotherm, an alternative method of measuring macropore diameters has been devised. Macropores are arbitrarily defined as pores greater than 1000 Å in diameter. The sample is evacuated and the volume of mercury which penetrates the carbon at varying pressures, is recorded. By means of the "capillary-rise" equation,

\[ D = \frac{4 \sigma \cos \theta}{P} \]

Where
- \( \theta \) = carbon-mercury contact angle
- \( \sigma \) = surface tension of mercury
- \( P \) = applied pressure,

the recorded pressures are converted into pore diameters. The range normally covered is from 106,000 Å, the pore diameter into which the mercury will penetrate at atmospheric pressure, down to 1000 Å, the pore diameter into which mercury will penetrate at 2,000 psi. If desired, however, the range may be extended to smaller diameters by application of higher pressures and thus overlap the micropore size distribution obtained from the water isotherm.

The contribution of macropores to surface area and adsorptive capacity is very small. For this reason, the presence of macropores in carbon may in certain cases be a distinct disadvantage since extraneous pore volume results in unnecessary loss of density. However, in applications where diffusion is a factor controlling rate of adsorption, e.g., in adsorption from solution, large macropores may well be an essential feature of carbon structure.

D. THE PORE SIZE DISTRIBUTION

1. Total Pore Volume

Since mercury penetration data show only change in volume with pore diameter, the total pore volume of the carbon, as outlined by mercury at atmospheric pressure, must be obtained in order to fix the upper limit of the complete pore size distribution. This is accomplished by two measurements.

a. The volume of mercury which the sample displaces is measured and represents the volume of the carbon granules. When this measurement is made at atmospheric pressure, only the largest pores, those greater than 106,000 Å (which are mainly the result of surface roughness of the particles), are filled with mercury, just as at the start of the mercury penetration measurement.

b. The volume of helium which the sample displaces is measured. Since helium penetrates into the smallest pores, the volume measured is that of the carbon skeleton. The difference in volume is then the total pore volume. By deducting the successive volume increments measured by mercury penetration from the total pore volume, the macropore size distribution may be plotted.

2. Pore Size Distribution

Between 10 and 1000 Å, therefore, the pore size distribution is calculated from the water isotherm; and between 1000 and 100,000 Å (.1-10 microns) from mercury displacement, helium displacement and mercury penetration data. Since two different theoretical concepts...
are involved in the determination of the complete curve, the smoothness with which both halves extrapolate into each other is a measure of the validity of the technique as a whole. Surprisingly good closure of the curves is found in most cases, considering inaccuracies which may be introduced in sampling or in the determination of pore diameters in the upper portion of the water isotherm. The salient features are illustrated in Figure 7.

3. Surface Area—Pore Diameter Curves

If pores are regarded as being split up into cylindrical segments of fairly uniform diameter, and the pore volume associated with any particular segment is determined from the pore size distribution, it is then possible to determine the area of the walls of the segment through simple geometry by use of the relationship.

\[ \Delta V = \pi r^2 \Delta h \]

or \[ \Delta A = \frac{4 \Delta V}{D} \]

The areas of all the segments may be added or integrated starting with those of large diameter, and the figures obtained then represent the cumulative area of all the pores greater than the particular pore diameter down to which the summation is carried. The cumulative surface areas are plotted as a function of pore diameter.

The resulting curve is of great value in predicting capacities for different adsorbates by correlation with the surface area in pores greater than a specified diameter. Typical curves corresponding to the isotherms in Figures 3, 5 and 6 are shown in the accompanying sketch, Figure 9, it being understood that Figure 5 was the isotherm of a hypothetical decolorizing carbon.

A. MOLECULAR SCREENING—CONCEPT OF "AVAILABLE" SURFACE

If the chemical nature of the surface is regarded as playing a secondary role in adsorption, the adsorptive properties of activated carbon can be attributed mainly to surface area and pore structure.

It is apparent from pore size distribution data that the major contribution to surface area is located in pores of molecular dimensions. It seems logical to assume that a molecule, because of steric effects, will not readily penetrate into a pore smaller than a certain critical diameter and will be excluded from pores smaller than this; hence, the concept that molecules are "screened out" by pores smaller than a minimum diameter which is a characteristic of the adsorbate and related to molecular size. Furthermore, for any molecule the effective surface area for adsorption can exist only in pores which the molecule can enter.

Figure 10 attempts to illustrate this concept for the case in which two adsorbate molecules in a solvent (not shown) compete with each other for adsorbent surface. Because of the irregular shape of both pores and molecules and also by virtue of constant molecular motion, the fine pores are not blocked by the large molecules but are still free for entry by small molecules. As a contributing factor, the greater mobility of the smaller molecule should permit it to diffuse ahead of the larger molecule and penetrate the fine pores first.

This concept of "available" surface, that is, surface area accessible to the adsorbate molecule, when applied in conjunction with the surface area-diameter curves referred to in Section II, provides a powerful tool by means of which apparently unrelated adsorptive properties of activated carbons may be satisfactorily accounted for. However, in the event that critical comparisons of different carbons are to be made on this basis, it is desirable that secondary factors, such as the chemical nature of the surface, be held as constant as possible either by appropriate treatment of the carbons concerned or by specifying uniformity in raw material and method of manufacture of the carbons to be compared.
CONCEPT OF MOLECULAR SCREENING IN MICROPORES

Figure 10
ELECTRON MICROSCOPE PHOTOGRAPH OF A PITTSBURGH ACTIVATED CARBON

While the artist's conception on the preceding page shows a cross-sectional view, the above illustration is an actual photograph. Light areas are pure openings. Dark areas represent the carbon skeletal structure.
B. CORRELATION OF ADSORPTIVE PROPERTIES AND PORE STRUCTURE

The adsorptive properties of activated carbon are usually expressed in terms of its capacity for a given adsorbate at a specified equilibrium concentration. The adsorbates most often used to characterize carbon are colored bodies or dyes, such as iodine, methylene blue and molasses; and the respective capacities are referred to as iodine number, methylene blue number and so on. To illustrate how adsorbate numbers are correlated with pore structure, the surface area in pores greater than various pore diameters, determined from surface area-pore diameter curves, is tabulated for a set of carbons whose iodine numbers, for example, have been determined. (Table 2)

<table>
<thead>
<tr>
<th>Carbon</th>
<th>Iodine No.</th>
<th>Area in pores greater than 8 Å</th>
<th>9 Å</th>
<th>10 Å</th>
<th>11 Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1262</td>
<td>1118</td>
<td>1110</td>
<td>1110</td>
<td>1087</td>
</tr>
<tr>
<td>B</td>
<td>1053</td>
<td>1032</td>
<td>1015</td>
<td>985</td>
<td>955</td>
</tr>
<tr>
<td>C</td>
<td>1053</td>
<td>885</td>
<td>870</td>
<td>857</td>
<td>841</td>
</tr>
<tr>
<td>D</td>
<td>911</td>
<td>844</td>
<td>815</td>
<td>787</td>
<td>760</td>
</tr>
</tbody>
</table>

The area in pores greater than a selected pore diameter is then plotted as a function of iodine number for each carbon, and this procedure is repeated for different pore diameters until a straight line passing through the origin and all points is obtained. In the normal case, the best straight line through the points is drawn and that line is selected which gives the minimum standard deviation of the points. (Figures 11 to 13)

The following results have been obtained using this technique.

<table>
<thead>
<tr>
<th>Adsorbate</th>
<th>Minimum Pore Diameter, Å</th>
<th>Equation of Line</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iodine</td>
<td>10</td>
<td>y = 4.8 + 0.30X</td>
</tr>
<tr>
<td>Potassium permanganate</td>
<td>10</td>
<td>y = 0.4 + 0.34X</td>
</tr>
<tr>
<td>Methylene blue</td>
<td>15</td>
<td>y = 17 + 0.30X</td>
</tr>
<tr>
<td>Erythrosine red</td>
<td>19</td>
<td>y = 129 + X</td>
</tr>
<tr>
<td>Molasses</td>
<td>28</td>
<td>y = 17.1 + 3.07X</td>
</tr>
</tbody>
</table>

The correlations are shown in Figures 14 to 18.

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All of the above molecules were adsorbed from aqueous solutions where the pH was essentially neutral. If adsorption is carried out in solutions of varying pH, considerable discrepancy in capacity may be anticipated, since pH affects both the solubility and ionic character of the adsorbate. It is therefore necessary to exert caution when predictions of structure from adsorptive properties are attempted, unless there is reason to believe that the pH of the solutions in contact with the carbon is fairly constant.

Once the minimum pore diameter for a particular adsorbate has been established, the capacity of other carbons for this adsorbate may be predicted from their surface area-diameter curves. Conversely, if several adsorbate numbers are known for a particular carbon, the surface area in pores greater than the minimum pore diameters previously established for these adsorbates may be predicted. Providing that the adsorbates selected have minimum pore diameters which are well-spaced and cover a fairly wide range, say 10-40 Å, it is possible by this means to reconstruct the surface area-diameter curve for a carbon whose pore size distribution is unknown.

For example, given a carbon with the following adsorbate numbers, and applying the equations in Table 3, surface areas are then tabulated as follows:

<table>
<thead>
<tr>
<th>Adsorbate</th>
<th>Adsorbate Number</th>
<th>Area in pores greater-than</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iodine</td>
<td>1262</td>
<td>1163</td>
</tr>
<tr>
<td>Methylene blue</td>
<td>360</td>
<td>1060</td>
</tr>
<tr>
<td>Erythrosine red</td>
<td>240</td>
<td>743</td>
</tr>
<tr>
<td>Molasses</td>
<td>400</td>
<td>291</td>
</tr>
</tbody>
</table>

The resulting surface area-diameter curve is plotted in Figure 19, and the dotted line shows the curve obtained from the pore size distribution.

The application of structure correlations to other adsorbate systems has shown that streptomycin adsorption correlates with surface area in pores greater than 18 Å in diameter (Figure 20), and nylon salt adsorption with surface area in pores greater than 20 Å (Figure 21).

- **Figure 20**
  - Streptomycin Capacity
  - Surface Area in pores > 18 Å

- **Figure 21**
  - Nylon Salt Capacity
  - Surface Area in pores > 20 Å

A more rapid but less accurate method of correlating structure is to plot adsorbate numbers as a function of other adsorbate numbers whose minimum diameters are already established. By this means, aniline adsorption is shown to correlate with iodine and methylene blue numbers for a minimum pore diameter of 10-15 Å (Figures 22 and 23).

- **Figure 22**
  - 60 Aniline No.
  - Iodine No.
Cyclohexylamine adsorption apparently correlates with iodine number for a minimum pore diameter of 10Å.

Two other bits of evidences favoring the molecular screening concept are:

1. The development of adsorptive capacity for successively larger molecules as activation proceeds, on the assumption that activation effects the progressive enlargement of small pores.

2. The separation of gases and vapors of varying molecular size by the process of selective adsorption by so-called "molecular sieves".

C. ADSORPTION OF MIXTURES

When more than one adsorbate is present in solution, it is inevitable that there will be competition for available surface. In the absence of molecular screening, the available surface will be identical for all adsorbates concerned. However, if molecular screening occurs, the available surface may not be the same for each molecule, in which case preferential adsorption of a particular adsorbate within a narrow pore size range is a possibility.

For example, the adsorption of hydroxymethyl furfural (HMF) in aqueous solution correlates with surface area in pores greater than 15 Å, and adsorption of protein and color bodies with surface area in pores greater than 23 Å. However, when adsorption of HMF takes place in the presence of protein and color bodies, it is necessary to postulate a maximum, as well as a minimum, pore diameter in order to effect satisfactory correlation. HMF adsorption has been correlated with surface area in pores between 15 and 23 Å, and it therefore appears that adsorption in larger pores is restricted by competing protein and color bodies, as illustrated by analogy in Figure 10.

D. ENERGY OF ADSORPTION

When a gas or vapor is adsorbed on the surface of activated carbon, latent heat is released in a similar manner as when steam condenses or water turns to ice. In most cases, the heat of adsorption (E₁) is greater than the heat of liquefaction (E₂) of an equivalent amount of vapor, that is, E₁ > E₂. The difference, E₁ - E₂, is referred to as the net heat of adsorption. When E₁ - E₂ is positive, the gas isotherm is concave to the pressure axis at low pressures; when it is zero, the isotherm is linear and when it is negative, the isotherm is convex as in the case of the water isotherms in Section II. The instantaneous heat of adsorption approaches the heat of liquefaction as saturation is reached. Heat of wetting is equal to the overall heat evolved on adsorption of a vapor at saturation less the heat of liquefaction of the quantity of liquid adsorbed by the carbon at saturation.

It has been observed from pure gas isotherms that certain fine pore carbons adsorb ethylene at low concentrations to a greater extent than can be accounted for by comparison of their total surface areas as measured by nitrogen adsorption with those of other carbons. Furthermore, it has been observed* that in air activation of a lignin carbon, the adsorptive power for phenol develops during the initial stages but does not continue to increase during subsequent stages, even though the total surface area is increasing. Such phenomena cannot be explained by the basis of molecular screening but can be explained on the basis of pore structure by means of a corollary concept that the surface is non-uniform with respect to adsorption and that fine pores enclose high energy sites and large pores low energy sites of adsorption.

The energy distribution of activated carbons, calculated from data on desorption of ethyl chloride vapor, shows that the energy of different sites on the surface varies over a wide range. If these energy distributions are compared with surface area-pore diameter distributions obtained by differentiating cumulative surface area-pore diameter curves, there is a striking similarity. It is possible to correlate energy of adsorption with pore diameter; and the evidence supports the belief that the smaller the pore diameter, the higher the energy of the sites within the pore. In a pore of molecular dimensions, the molecule is probably attracted by surface forces on several sides; that is, there may be a cooperative wall effect assisting adsorption. It is reasonable to suppose that adsorbate molecules will be attracted first to regions of the surface which have the highest energy. Consequently, at low concentrations, high energy surface area in fine pores may be the dominant factor determining equilibrium capacity rather than total surface area in all pores. The possibility that very fine pores could result in looser packing of the molecules would lead one to suspect that there is probably an optimum diameter for each adsorbate which will give maximum adsorption.

*J. W. Hassler, Abstract of paper presented at September, 1932, ACS meeting, Atlantic City, New Jersey.

It should be pointed out that the presence of considerable high energy surface in activated carbon may not always be advantageous. Strong adsorption in fine pores is associated with high retenivity, the ability to retain the adsorbed layer under specified conditions favoring desorption. In cases where solvent vapors already adsorbed must be recovered for economic reasons, it may be correspondingly more difficult to accomplish this if a fine pored carbon is employed.

E. THE PREDICTION OF ADSORPTION ISOTHERMS

Although satisfactory techniques are available for determining the pore size distribution of an activated carbon from either its water or nitrogen desorption isotherm, it is not yet possible to predict adsorption isotherms from the pore size distribution alone. Nevertheless, the generalized adsorption correlation of Lewis et al., which is based on the polanyi adsorption potential theory, can be used to investigate the relationship between filled pore volume and adsorption-free energy. It has been remarkably successful in correlating the adsorption isotherms of a large number of gases and vapors on a single activated carbon, and in the absence of experimental data, its use is recommended for preliminary estimation of the adsorption capacity of gases.

Grant, Manes, and Smith, A. I. Ch. E. Journal, 8, 403 (1962).
Appendix G-2

Laboratory Evaluation of
Granular Activated Carbon for
Liquid Phase Applications
Calgon Corporation
The laboratory evaluation of granular activated carbons for liquid phase applications
Introduction

Although regenerable granular activated carbons have been employed for a number of years for the removal of soluble adsorbates from liquids, most of the published data in the field of liquid phase activated carbon adsorption apply only to pulverized carbon in batch systems. A sound method for predicting the actual or process performance of granular carbon in columns from batch data is not yet available. Consequently, until valid mathematical data can be established, each new application for granular carbon must be considered individually.

This discussion is primarily concerned with describing the laboratory procedures that we have found most valuable for obtaining isotherm and preliminary column design data for the use of granular activated carbon in liquid phase applications.

Whether granular activated carbon is considered for a new application or for the replacement of pulverized carbon, it is essential to determine its efficiency and related advantages for the specific application. A complete laboratory investigation would generally consist of two parts. First, preliminary isotherm tests would be performed to demonstrate the feasibility of granular carbon treatment. Second, laboratory column tests would be conducted to obtain data to be used in designing the full-scale plant.

Depending on the amount of background information available, the preliminary tests may be unnecessary in some cases. Assume, however, for the sake of discussion, that we are dealing with an application about which little is known, and consider first the preliminary investigation.

Definition of terms used here is in accordance with ASTM Standard Definitions of Terms Relating to Activated Carbon—Designation: D2652

Adsorption Isotherms

THEORY

Inspection of an adsorption isotherm is a relatively simple method of determining the feasibility of using granular activated carbon for a particular application. A liquid phase isotherm shows the distribution of adsorbate (that which is adsorbable) between the adsorbed phase and the solution phase at equilibrium. It is a plot of the amount of adsorbate adsorbed per unit weight of carbon versus the concentration of adsorbate remaining in solution.

Generally, straight line plots can be obtained by making use of the empirical Freundlich equation which relates the amount of adsorbate in the solution phase to that in the adsorbed phase by the expression:

\[ \frac{x}{m} = k c^{1/n} \]

Where

- \( x \) = amount of adsorbate adsorbed
- \( m \) = weight of carbon
- \( x/m \) = concentration in the adsorbed phase (i.e., the amount of adsorbate adsorbed per unit weight of carbon).
- \( c \) = equilibrium concentration of adsorbate in solution after adsorption
- \( k \) and \( n \) are constants.

Taking the logarithm of both sides we obtain:

\[ \log \frac{x}{m} = \log k + \frac{1}{n} \log c \]

This is the equation of a straight line whose slope is \( 1/n \) and whose intercept is \( k \) at \( c = 1 \). Therefore, if \( x/m \) is plotted against \( c \) on log-log paper, a straight line should normally be obtained. However, there are occasions, that will be explained later, where this is not true. The straight line and the occasional curved isotherm lines provide valuable predictive information for adsorption operations.

EXPERIMENTAL

Data for plotting isotherms are obtained by treating fixed quantities of the liquid to be tested with a series of known weights of carbon. The carbon-liquid mixture is agitated for a fixed time (previously determined from contact time study) at a constant temperature. After the carbon has been removed from the liquid by a suitable means of separation, the residual adsorbate in solution is determined. From these measurements, all of the values necessary to plot an isotherm can be calculated.

Selection of Experimental Conditions

Particle Size of the Carbon

In a liquid phase application, the transfer of the adsorbate from the bulk solution to the carbon particle must proceed through at least two stages:

1. Transfer of the adsorbate from the bulk liquid to the surface of the carbon particle.
2. Migration of the adsorbate from the surface of the carbon to the adsorption site within the particle.

Kinetic experiments demonstrate that Step 1 is appreciably more rapid than Step 2; so the latter is normally the primary rate-determining step. The rate of adsorption, then, will vary with the diameter of the carbon particles used in the test.

In order to increase the rate of adsorption, and thus decrease the time necessary to complete the isotherm, it is recommended that the granular carbon be pulverized so that 95 wt.% will pass through a 325 mesh screen. Contrary to what one might think, this pulverization does not significantly increase the surface area. In fact, the increase in the surface area is less than 1% since the vast majority of surface area is contributed by pore walls rather than external surface.

Temperature of the Liquid

Adsorption efficiency is generally a function of the system temperature. For laboratory evaluation of granular activated carbon for liquid phase applications, it is recommended that the temperature of the existing process stream be investigated first. If the desired degree of adsorption occurs at this temperature, then the plant operating parameters will not have to be altered. If, on the other hand, the desired degree of adsorption does not take place at the existing process temperature, then either a higher and/or lower temperature
should be investigated. The selection of the temperature will depend upon one or more of the following:
 a) viscosity;
 b) thermal stability characteristics of the test liquid; and,
 c) feasibility of changing the process temperature in the plant.

**pH of the Liquid**

Adsorption capacity can also be a function of the pH of the liquid. Normally, when performing laboratory evaluations, the pH of the process stream is used. If the desired degree of adsorption does not take place at the process pH, then various pH levels should be investigated. Care must be taken when adjusting the pH of a process stream to make sure that the change in pH does not degrade or decompose the particular product.

When treating liquids with a low pH, it is advisable to use an acid-washed carbon such as Calgon® Type CPG, since a portion of the ash constituents of non-acid-washed activated carbon will be solubilized under acidic conditions.

**Contact Time for Isotherm**

Contact time is very critical to the adsorption process. It should be sufficiently long to allow an approach to adsorption equilibrium. A preliminary experiment should be performed to determine the contact time required to perform an isotherm that reaches equilibrium.

One-half gram portions of the pulverized activated carbon should be added to several 100 gram portions of the test liquid (when the density of the liquid is near that of water, 100 ml of the liquid, carefully measured, may be used) and each portion agitated for various time periods at the process temperature and pH. For example, portion 1 is agitated for ½ hr., portion 2 for 1 hr., portion 3 for 2 hrs., and so forth. At the end of each contact time, the carbon is removed by a suitable means such as filtration or centrifugation. If the quantity of adsorbate remaining in solution is plotted as a function of time, a curve similar to Figure 1 should be obtained. It is apparent from inspection of Figure 1 that the contact time required to reach equilibrium for this particular liquid is 3 hours.

**Carbon Dosage**

In order to obtain a meaningful isotherm, as wide a range of carbon dosages as practical should be used. Recommended dosages are 0.05, 0.1, 0.2, 0.5, 1.0, 2.5, 5.0, and 10.0 grams of carbon per 100 grams of the test liquid.

**Apparatus**

Pulverized carbon for isotherm testing can be obtained from Calgon. If suitable mechanical equipment, such as laboratory ball mill, is available and it is desired to pulverize carbon for the isotherm test, it must be pulverized so that 95 wt.% passes through a 325 mesh screen.

Stoppered flasks or pressure bottles are satisfactory containers for the carbon-liquid mixture. Manual agitation, even at frequent intervals, is often inadequate; therefore, a mechanical shaker is highly desirable. If a constant temperature bath is used, the design of the shaker should permit immersion of the containers so that the liquid temperature is maintained at the desired value. A wrist-action type shaker has proven to be quite adequate in our laboratories.

**Carbon-Liquid Separation**

Prior to analysis of the treated liquid, the carbon must be removed from the liquid. Generally, the carbon can be removed by filtration through a 0.45 micron pore size filter pad. In some cases, a smaller pore size filter pad may be necessary to remove all of the carbon particles. The filter pad which is used must be compatible with the treated liquid. The filtration rate may be increased by using vacuum or pressure.

If the liquid is so viscous that filtration is difficult, centrifugation can sometimes be used to effect a suitable separation.

**Determination of Adsorbate Removal**

After the carbon has been removed from the treated liquid, the amount of adsorbate remaining in solution must be determined. Any analytical method or instrument that can determine the quantity of the particular adsorbate remaining in the test liquid may be used. Typical techniques are filtration, spectrophotometry, gas chromatography, and total organic carbon analysis.

**General Procedure for Isotherms**

1. **Pulverize a representative sample of the granular carbon (a 25 - 30 gram sample is usually adequate) so that 95 wt.% will pass through a 325 mesh screen, or use pulverized material supplied by Calgon. Oven-dry the pulverized sample for three hours at 150°C.**

2. **Obtain a representative sample of the liquid to be tested. Any suspended matter should be removed by filtration.**

3. **From the oven-dried pulverized sample, weigh out the carbon samples on an analytical balance and transfer the weighed samples to suitable containers. Sample weights should be chosen in accordance with the suggestions given under Carbon Dosage.**

4. **Weigh 100 grams of the test liquid into each container. After manually swirling to make certain all the carbon is wetted, clamp each container on the shaker. Also weigh 100 grams of the test liquid into a container without carbon (this will serve as the "blank" or control sample) and place it on the shaker with the other containers. Agitate all the containers, at the desired temperature, for the contact time that was determined from the contact temperature rate study, or as suggested by the Calgon Technical Sales Representative.**

5. **After the required contact time has elapsed, separate the carbon from the liquid by an appropriate method in accordance with the suggestions given under Carbon-Liquid Separation. The control sample should be subjected to the same separation procedure as the other samples.**

6. **Determine the residual adsorbate in solution for each of the samples by the appropriate method for detecting that particular material.**

7. **Tabulate the data as shown in Table 1. The residual adsorbate concentration (c) is obtained directly from analysis of the treated solutions. The control sample adsorbate concentration (c<sub>0</sub>) is obtained by analysis of the control solution. The amount of adsorbate adsorbed (x) is obtained by subtracting the value of c from that of c<sub>0</sub>. Divide x by the weight of carbon used (m) to obtain the quantity (x/m) which is the amount of adsorbate adsorbed per unit weight of carbon.**

8. **On log-log paper, plot the values of c on the horizontal axis against those of x/m on the vertical axis and draw the best straight line through the points as illustrated in Figure 2.**
Interpretation of the Isotherm

From an inspection of the isotherm, it can be determined whether or not the desired degree of adsorbate removal is possible with the particular activated carbon tested. In addition, other information can be obtained from a more detailed examination of the isotherm plot.

Calculation of Adsorptive Capacity

If a vertical line is drawn from the point on the horizontal axis corresponding to the influent concentration ($c_0$) line, and the best line through the data is extrapolated to intersect this ($c_0$) line, the ($x/m$) value at the point of intersection can be read from the vertical scale. This ($x/m$) value represents the amount of adsorbate adsorbed per unit weight of carbon when that carbon is in equilibrium with the influent concentration. This represents the ultimate capacity of the carbon at these conditions. This value may or may not be obtained in column operation, since the rate of adsorption, and thus the superficial contact time in a column system, is important.

From the value of ($x/m$) the quantity of liquid treated can be calculated using the following formula:

$$W_{co} = \frac{(x/m)_{co} (W)}{c_0}$$

Where $W_{co}$ = theoretical weight of liquid treated per gram (or unit weight) of carbon.

$$(x/m)_{co} = \text{capacity per gram (or unit weight) of carbon at the influent concentration.}$$

$w = \text{weight of liquid used in the isotherm test.}$

$C_0 = \text{influent concentration.}$

If the liquid was measured by volume, the same calculation should be used by substituting $V_{co}$ and $V$ in the proper places.

$$V_{co} = \frac{(x/m)_{co} (V)}{c_0}$$

Where $V_{co}$ = theoretical volume of liquid treated per gram (or unit weight) of carbon.

$$(x/m)_{co} = \text{same as (1)}$$

$V = \text{volume of liquid used in the isotherm test.}$

$C_0 = \text{same as (1)}$

Here is an Example of a Capacity Calculation:

From the example in Table 1:

$$W_{co} = \frac{(x/m)_{co} (W)}{c_0}$$

$$(x/m)_{co} = 26.5$$

$C_0 = 18.15$

For complete adsorbate removal:

$$W_{co} = \frac{(x/m)_{co} (W)}{c_0} = 26.5 \cdot \frac{100 \text{ gm}}{18.15} = 146 \text{ gm liquid per gm carbon}$$

From these figures, the theoretical granular column dosage (or usage rate) to treat the liquid may be obtained. In the example, the grams of carbon required to treat one gram of solution is:

$$\frac{1}{146} = 0.00685$$

In this instance 0.685 lbs. of carbon would theoretically treat 100 lbs. of liquid. However, in most column systems the carbon dosage will be greater than this because the carbon which is removed from the system will not be saturated.

Comparison of Different Carbons

The performance of two or more carbons in the same application can be compared by examining isotherms run as outlined previously. Usually, the granular carbon with the higher ($x/m)_{co}$ value would be preferred for the application.
Non-Linear Isotherms

It was previously stated that, ideally, straight line isotherm plots are obtained. However, there may occasionally be departures from linearity.

A curve similar to that shown in 3A may be obtained if a nonadsorbable impurity is present in the liquid being treated. Subtracting \( c_i \) from \( c_0 \) and plotting the isotherm will usually yield a straight line.

A sudden change in slope (illustrated in 3B) would indicate two components (adsorbates) are present which are adsorbed at different rates and markedly different capacities. The plot in 3C, similar to 3B, illustrates a situation in which at least 3 compounds (adsorbates) are present, and all are adsorbed differently. Of course, when many adsorbate species are present — e.g., undefined "color" — but when all are adsorbed similarly, straight line isotherms will still be obtained.

The curve shown in D indicates that the adsorbate has reached its maximum surface coverage at capacity \( X \).

**Figure 3 — Non-Linear Isotherms**

```
A
B
C
D
```

Summary

From an isotherm test one can determine whether or not a required adsorbate removal can be accomplished, and can obtain an approximation of the ultimate capacity of the granular carbon for that application. From the capacity figure an estimate of the minimum granular carbon usage rate necessary to meet the treatment objective can usually be obtained.

Isotherm tests also afford a convenient method for comparing different carbons and for investigating the effects of \( \text{pH} \) and temperature.

The next step in evaluating granular activated carbon is dynamic laboratory column tests.

**Adsorption Studies In Columns**

In preparation for plant scale operations, additional information that is not available from the isotherm must be obtained. The operating capacity as well as the flow rate, mass transfer zone, and contact time must be established in order to determine the number and size of the columns necessary for continuous treatment.

**APPARATUS**

**Columns**

In laboratory column testing, it is recommended that fifty (50) linear feet of carbon be used in a minimum of five (5) individual columns; however, ten columns are preferred since this would provide more data. The diameter of the columns should be as large as is practical, with a minimum diameter of one inch. The columns may be constructed of Pyrex-type glass, metal, or plastic. Care should be taken to choose a material of construction that is compatible with (1) the liquid being treated, (2) the pressure, and (3) the temperature of the system. If the column testing is to be performed at elevated temperature, water or steam jackets, electrical windings, infrared lamps, or an oven can be used.

**Connections**

The material of construction for tubing and fittings must be compatible with the liquid being investigated and the test conditions being used.

**Pump**

A small pump capable of delivering uniform rates, at the desired flow and pressure is required.

**Figure 4 — Laboratory Columns**

**Arrangement of Equipment**

The columns are mounted vertically and connected in series as shown in Figure 4. Provisions must be made for taking samples between each column so that adsorption can be studied as a function of bed depth, flow rate, and volume of liquid treated. The carbon bed is contained at the top and bottom of each column by a stainless steel screen, glass wool, porous plate, or filter screen material. Pressure gauges should be installed at various points in the system. For more detailed equipment specifications contact the Calgon Technical Sales Representative in your area.

**PROCEDURES**

**System Preparation**

The amount of granular activated carbon required to fill each column may be determined as follows:

\[
\text{Weight carbon/column} = V \times \text{A.D.} \times 0.85
\]

Where:  
- \( V \) = Volume of column  
- \( \text{A.D.} \) = Apparent Density of carbon

The required amount of granular carbon for each column should be weighed into a suitable container and degassed or wetted by using one of the following procedures:

A. Boil in water for a minimum of 2 hours.
B. Soak in water for a minimum of 24 hours.
C. Soak in purified test liquid for a minimum of 24 hours.
D. Boil or heat at elevated temperature in a compatible solvent for a minimum of 2 hours.
E. Soak in a compatible solvent for a minimum of 24 hours.

The degassed carbon, as a slurry, is charged to the columns in small increments, keeping a thin layer of supernatant liquid above the carbon during charging. This is best accomplished by filling the column one-third full with the degassing or wetting liquid before starting to charge the carbon slurry. This charging procedure should be repeated for each column. Prior to connecting the columns in series, all connecting tubing and other void space must be filled with liquid in order to avoid the formation of gas pockets in the carbon bed. Any gas build-up can be released through the vents at the top of the columns.

**NOTE:** In some cases it may be necessary to de-aerate the liquid used for degassing or wetting the carbon (See below "Liquid Pretreatment").

**Column Operation**

**Temperature**

Adsorption capacity is a function of diffusion rate; and, since this is affected by viscosity, the columns should be operated at the plant process or isotherm temperature to eliminate this variable.
Liquid Pretreatment

If the isotherm investigation indicated that a pH adjustment was necessary before carbon treating, then the same pH adjustment should be made to the liquid before granular carbon column studies. In cases where suspended matter is present in the test liquid, it should be removed by filtration before the liquid is pumped to the columns. If the test liquid has been stored at a low temperature for preservation purposes, subsequent heating to room temperature, or higher, may result in degassing the liquid. When these conditions exist, the liquid must be deaerated prior to pumping it through the carbon columns.

Flow Rate

The feed rate to be used depends upon the diameter or cross-sectional area of the tube which was selected for column testing. For preliminary trials a feed rate of 2 gpm/ft.² of cross-sectional bed area or 4.9 M/hr. is recommended. If sufficient time and test liquid are available, feed rates of 1 and 3 gpm/ft.² of cross-sectional area should also be investigated in order to determine the best flow rate for the system. For column diameters normally used in laboratory work, a rate of 2 gpm/ft.² is equivalent to the rates given in Table 2.

Duration of Column Testing

Ideally, the test should be continued until the first column is saturated and the specification value of adsorbate in the liquid has been detected in the effluent from the last column in the series. If time and/or the quantity of test liquid available does not permit the test to be run to ideal completion, it should be run until breakthrough occurs in the last column, or until the first two or three columns are saturated.

Displacement, Sampling and Analysis

So that accurate breakthrough curves can be defined, the wetting liquid must be displaced from the columns in order to establish a treatment starting point for each column used in the study. The test liquid is pumped through the column system until the wetting liquid has been displaced and only the test liquid is in contact with the carbon beds. This displacement process is monitored by analyzing the effluent from each column. The volume of the samples taken during this process should only be the quantity necessary to determine the concentration (i.e. that quantity required by the analytical method). When the effluent liquid from the first column is the same as the test feed liquid, then it can be assumed that most of the wetting liquid has been displaced from that column. A suggested record keeping procedure is shown in Exhibit 1. After the wetting liquid is displaced from the first column, the collection of data to determine the breakthrough curve can be initiated. The breakthrough curve is determined by sampling at intervals and analyzing for adsorbate, and by measuring and recording the volume throughput. (Refer to Exhibit 2 for a suggested data recording format).

The displacement procedure is repeated for each column until the wetting liquid from every column has been displaced and the initial adsorbate breakpoint determined. The breakthrough curve for each column must then be defined as previously described. Sampling should be frequent enough

### Exhibit 1

**RECORD OF DISPLACEMENT PROCESS**

<table>
<thead>
<tr>
<th>TIME</th>
<th>CONCENTRATION OF TEST LIQUID</th>
<th>CONCENTRATION OF TEST LIQUID IN COLUMN EFFLUENT</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td>T₁</td>
<td>100</td>
<td>0 No Sample</td>
<td></td>
</tr>
<tr>
<td>T₂</td>
<td>100</td>
<td>10 No Sample</td>
<td></td>
</tr>
<tr>
<td>T₃</td>
<td>100</td>
<td>30 No Sample</td>
<td></td>
</tr>
<tr>
<td>T₄</td>
<td>100</td>
<td>50 20 No Sample</td>
<td></td>
</tr>
<tr>
<td>T₅</td>
<td>100</td>
<td>100 45 No Sample</td>
<td>Col. A displaced at time = t₅</td>
</tr>
<tr>
<td>T₆</td>
<td>100</td>
<td>70 20 No Sample</td>
<td></td>
</tr>
<tr>
<td>T₇</td>
<td>100</td>
<td>100 40 No Sample</td>
<td>Col. B displaced at time = t₇</td>
</tr>
</tbody>
</table>

### Exhibit 2

**Type of Carbon**

**Flow**

**Plant Name**

**Column No.**

**Weight of Carbon**

**Volume of Carbon**

**Contact Time**

### Table 2

**TUBE DIAMETER**

<table>
<thead>
<tr>
<th>Inches</th>
<th>Centimeters</th>
<th>cc./min.</th>
<th>gph</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>254</td>
<td>41.6</td>
<td>0.659</td>
</tr>
<tr>
<td>2</td>
<td>508</td>
<td>165.2</td>
<td>2.62</td>
</tr>
<tr>
<td>3</td>
<td>762</td>
<td>374.0</td>
<td>5.93</td>
</tr>
<tr>
<td>4</td>
<td>10.16</td>
<td>661.6</td>
<td>10.49</td>
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</table>
to establish the profile of the breakthrough curve for each column. Generally, the first column requires a high frequency of sampling, initially. In any event, a careful measurement and record of the volume put through each column must be maintained. This record must account for sample volumes removed for analysis. (NOTE: Sample volumes should be the minimum quantity required for the analysis and should not exceed ten percent of a column bed-volume. If a larger quantity of sample is required for analysis, it is recommended that you contact your Calgon Technical Sales Representative.)

As the wetting liquid is displaced from each column, sampling for adsorbate analysis should begin. (NOTE: Liquid which is collecting for sampling does not pass through each succeeding column. Therefore, the number of samples which are taken should be limited so that the effect on adsorption in succeeding columns is minimized).

Treatment of the Data

If, for any single column of the system used in the experiment, the percent adsorbate remaining in the effluent is plotted as a function of liquid throughput, a curve is obtained. The shape of this curve will vary as a function of liquid being treated, linear flow rate, and carbon particle size. An example of a set of such curves is shown in Figure 5. In this example, each column is assumed to contain two meters of carbon weighing five kilograms.

The point at which adsorbate first appears in the effluent is termed the breakthrough. The part of the curve between the breakpoint and the point at which the concentration of adsorbate in the effluent is the same as in the influent is called the breakthrough curve.

During the adsorption cycle in a column, a portion of the inlet section of the bed will most likely become saturated with adsorbate. At the same point in time, the carbon near the outlet will be only partially loaded with adsorbate. The extent of saturation will depend on the system design and operating parameters. Between these two extremes lies a zone in which the adsorption is occurring. This area is sometimes referred to as the mass transfer zone or MTZ.

In the example shown in Figure 5, the MTZ is determined as follows: The specification value is an instantaneous value of 20% adsorbate in the effluent. The MTZ is determined by observing the amount of carbon between the 20% level.
two beds in series, a greater percentage of carbon in the first column will be saturated; and so, the dosage will be something between 0.25 and the isotherm dosage indicated as 0.15 in this example. Thus, judgement must be used in determining the dosage to be used for design purposes.

It should also be noted that this method of interpretation assumes that equal superficial contact time gives equal performance. In many cases this is only true when the linear velocities in the commercial unit are the same as in the pilot unit. When the velocities are 2 gpm/ft² (4.4 M/hr.) or less, it is generally true that somewhat shorter mass transfer zones are obtained as the linear velocity is increased. This is the opposite phenomenon than is observed at high rates. Therefore, if the commercial unit rate is more than 15 percent less than that used in the laboratory, there may be a detectable difference in the performance. For this reason, it is important to investigate, in the laboratory, the effect of different flow rates.

It should be emphasized that in many cases there will be alternate ways in which a system can be designed and operated. Unfortunately, short-cut methods for designing the optimum system are not available. Therefore, it is essential that the laboratory evaluation be conducted as thoroughly as possible. Also, the interpretation of the data must be correct. Your technical representative will welcome the opportunity to assist you in any way possible.

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Light areas are pore openings. Dark areas represent the carbon skeletal structure.
Appendix H

Guidelines for Column Operating Procedures
With Activated Carbon
Filtrasorb® granular activated carbon waste water treatment systems are capable of removing dissolved organic contaminants from municipal and industrial waste waters to levels beyond the upper limits that biological treatment can produce. These systems are available in various configurations to utilize the carbon bed volume most efficiently for specific requirements.

The following four basic designs have been employed for waste water treatment and/or water reclamation applications:

1. **FIXED BEDS IN SERIES**
   Fixed beds in series usually employ several adsorbers operated downflow in series. The lead adsorber in the series receives the influent waste water and removes the highest concentration of wastes. The treated effluent is then passed to the second, third, and succeeding units. In this way the concentration of contaminants becomes progressively less until the water is virtually free of contaminants. When the carbon in the lead unit becomes exhausted, it is taken out of service for reactivation and the influent load is transferred to the second unit and passed to the third and subsequent units. After reactivation, the lead unit is placed back into service at the end of the process. It then sequentially advances to the lead position, counter-current to the waste flow. This design is used where high quality effluent is desired. It requires backwash capabilities to remove accumulated solids.

2. **MOVING BEDS**
   A moving bed is a refinement of the counter-current operating principle. Waste water flow is directed upward through the bottom of the adsorber and clean water is drawn off at the top. In this system, the highest concentration of contaminants is adsorbed by the carbon at the base of the adsorber. The unit is designed to periodically remove slugs of exhausted carbon through the bottom for reactivation. This permits the bed to drop and allows for replenishing with reactivated carbon added at the top of the adsorber.

3. **FIXED BEDS IN PARALLEL**
   Fixed beds in parallel employ several adsorbers operating downflow which receive the influent water simultaneously. The carbon from each unit is replaced as a batch upon exhaustion. The start-up of the adsorbers is staggered so that exhaustion of the units can occur in sequence. Blending of highly purified effluent from newly reactivated units with that from partially exhausted units can prolong the useful life of each carbon batch. This technique reduces the frequency of reactivation and also produces an effluent that meets the treatment objective. This design favors large volume plants and requires backwash capabilities.
Fixed beds upflow-expanded employs adsorbers operated upflow in series or parallel. The result of the upflow operation expands the beds an additional 10% of the packed volume. The exhausted units are reactivated on a batch basis and rotated similarly to the downflow units in series or parallel operations. A suitable water distribution system with backwash capabilities is required for each adsorber in this design.

**Backwash Requirements**

With the exception of the moving bed adsorber which does not require backwashing, these adsorber designs require backwash and surface wash capabilities. In some cases, depending upon the concentration of suspended solids in the waste water, prefiltration may be desirable to reduce the solids load on the bed. However, whether or not prefiltration is provided, it is desirable to backwash the carbon beds for the following reasons:

1. To stratify the carbon bed.
2. To remove accumulated suspended matter.
3. To remove air pockets which can occur with certain water conditions.
4. To control bacterial growth in the carbon bed.

Mechanical features of the adsorbers associated with a backwash system include:

1. Adequate underdrain design to assure uniform water flow throughout the bed.
2. Freeboard between the top of the bed and the exit port to allow for bed expansion.
3. Surface wash nozzles to facilitate separation of suspended matter from granular carbon via scrubbing action.
thermal reactivation

A thermal reactivation system is recommended in a Filtrasorb waste water treatment system. It is used to reactivate exhausted carbon by volatilizing and oxidizing the adsorbed impurities in the carbon in the controlled atmosphere of a multi-hearth or rotary kiln furnace. The reactivated carbon is then quenched and transported as a slurry to the adsorbers for reuse. An average loss of about 5 per cent, which is replenished with virgin carbon, occurs for each reactivation cycle. (Reactivation equipment for granular carbon is described in Bulletin 20-5a and is available upon request.)

chemical regeneration

Recovery of valuable products after their adsorption on granular carbon is feasible in some instances. Removal of the product from the carbon can sometimes be accomplished with chemicals, solvents, and/or steam. Complete recovery is not always effected for each cycle and the carbon may require reactivation or replacement after a given number of recovery cycles. The equipment for these systems is dependent upon the waste stream and the product to be recovered.

design parameters

Adsorber Configuration .......... Fixed Beds in Series, Fixed Beds in Parallel, Moving Beds and Fixed Beds Upflow-expanded

Flow Rate—Maximum .................. 10 gpm/ft²
Recommended .......................... 5 gpm/ft²
Contact Time Function ................ 2-5 ft² of carbon per gpm of waste water
*For fixed bed upflow-expanded flow rate see chart.

FILTRASORB

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<td>Bed Expansion %</td>
<td>25-50</td>
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<td>Recommended Bed Expansion %</td>
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<td>Surface Wash</td>
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<td>Recommended</td>
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<tr>
<td>Head Loss</td>
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systems components

A separate brochure entitled “Hydraulic Transportation of Granular Activated Carbon” describes the equipment recommended for the transportation of granular carbon via water slurry. Additional information regarding screens, filter design, coatings, materials of construction, and other system components is available for specific application problems.

technical assistance

This bulletin contains only brief descriptions of the various activated carbon adsorption systems that are available for waste water treatment. A Calgon representative will be pleased to discuss your specific process. He can assist you in laboratory tests and in the set-up of a pilot-scale column in order to evaluate the feasibility of a granular activated carbon waste treatment system for your operation.
Wastewater treatment systems utilizing Filtrasorb carbon

The following illustrations show the flexibility of design configurations of some carbon wastewater treatment installations that are on-stream in municipal and industrial plants. These systems are custom designed into various configurations which can handle high volumes of wastewater and utilize minimal land area as compared to conventional types of wastewater treatment systems.

Upflow designed moving bed units employ Filtrasorb carbon to purify municipal wastewater from the resort community of Lake Tahoe, California. The purified wastewater is used to supply a nearby man-made lake. The lake covers 165 acres and contains a billion gallons of sparkling clear water that is being used for irrigation and recreational purposes.

These adsorber units employ granular activated carbon and are arranged to operate downflow in series. This installation is located in the community of Pomona, California. It purifies 0.3 million gallons of municipal wastewater per day.

A moving bed adsorber containing 50,000 lbs. of carbon is used to treat 500,000 gpd of dye rinse water for reuse at the Stephen-Leedom Carpet Company plant in Southampton, Pennsylvania. The facility was designed and constructed by Calgon Corporation on a turnkey basis. The system can be expanded to treat a million gallons per day by adding a second adsorber unit. This can be done on the present site.

Rhodia, Inc., Chipman Division in Portland, Oregon, is a herbicide manufacturing plant that employs granular activated carbon to remove phenolic compounds from its wastewater. The local sanitary authority had required the effluent from the Rhodia plant be limited to 1 mg/l phenol. Activated carbon was selected over five other processes based on effectiveness to remove phenolic compounds from water solution.

For further information write to Filtrasorb Department, Water Management Division, Calgon Corporation, P.O. Box 1346, Pittsburgh, Pa. 15230.
COLUMN OPERATING PROCEDURES WITH CALGON® GRANULAR ACTIVATED CARBON
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The Activated Carbon Division of Calgon Corporation, in pursuing the commercial application of its carbon products, has accumulated considerable engineering data pertaining to the design and operation of granular carbon systems. Each system, of course, is engineered to meet specific operating requirements within established economic latitudes. The material contained in this book pertains to the hydraulic transportation of granular carbon in a plant unit operation. The design guides herein provide sound engineering information that will promote trouble-free plant operation but are not universal in scope. These guides are based, necessarily, on the handling properties of Calgon Carbons only and do not apply to other granular products. For more detailed information and specific engineering data for your process requirements, contact the Activated Carbon Division of Calgon Corporation.
HYDRAULIC TRANSPORTATION OF GRANULAR CARBON

Hydraulic transportation in water or process liquors provides a clean, rapid, and economical means of conveying Calgon Granular Activated Carbon to and from adsorption columns. Many industrial installations, which are characterized by simplicity of operation, low maintenance and minimum investment, are now in operation. A schematic diagram of a typical system is shown in Figure 1.

Slurry Density
The density of carbon-water slurries varies with the method of transporting the carbon and, in some instances, with the viscosity of the liquid. These are some typical ranges:

\[
\begin{array}{ll}
\text{Method of Transporting} & \text{Pounds Carbon/ Gallon Water} \\
1. Eductors & 0.7 - 1.0 \\
2. Slurry Pumps & 1.0 - 2.0 \\
3. Pressure Pots & 3.0 - 4.0 \\
4. Gravity Through a Nozzle & 3.0 - 4.0 \\
\end{array}
\]

Slurry Velocity
The minimum velocity before saltation occurs depends on the liquid density and viscosity and the carbon particle size. In general, however, the design velocity in pipelines should be 3-7 feet per second. Higher velocities cause excessive attrition of the carbon.

HANDLING CHARACTERISTICS
Handling characteristics have been experimentally studied by using water slurries of Calgon Type CAL 12 x 40 Mesh Granular Carbon in a 2-inch pipeline. The following data were obtained:
Carbon Delivery Rates

Carbon delivery rate is a function of pipe diameter, slurry concentration and linear velocity. Data from pilot studies using 2-inch pipe are presented in Figure 2. These data were also used to calculate estimated performance in 1-inch pipe as plotted in Figure 3.
Pressure Drop

Pressure drop data for various slurry concentrations and velocities in 2", 4", and 6-inch pipe are presented in Figures 4, 5, and 6.
SLURRY PIPING AND VALVING

Piping which carries carbon-water slurries can be made of almost any material. Carbon steel is a common material which is used. Since wet carbon in contact with water causes corrosion, it is essential that the piping not have any dead-ends where carbon can “sit” in the line.

In order to minimize attrition there should be as few changes of direction as possible and bends should be long radius. Also, there should be no obstructions in the line.

There are basically three types of valves which have been used successfully in carbon slurry lines. These are:

1. Ball valves — regular and full-port.
   Material of construction — stainless.
2. Full flow Grinnell diaphragm valves.
3. Rubber pinch valves.

Since lines have been known to plug, there should be hose connection flush-out points every 10–20 feet in horizontal lines and at the bottom and top of vertical lines.

METHODS OF TRANSPORTING WATER-CARBON SLURRIES

Carbon slurries can be transported by using any of the following methods:

1. Water or Air Pressure (Blowcase)
2. Centrifugal Pump
3. Eductor
4. Others

The choice of motive power is a combination of customer preference, economics and differential head requirements. A brief description of each method follows:

1. Blowcase
   The blowcase uses air or water pressure applied in a specially designed pressure vessel to move the carbon slurry (see Figure 7). Carbon and water or process liquor are slurried in a feed tank located above the pressure vessel. The slurry falls into the pressure vessel and the valve is closed. Air or water pressure is applied and moves the slurry out through the discharge pipe. This method has found considerable success in commercial installations. If water is used as the transporting medium, a suitable lining on mild steel or stainless steel should be used to eliminate corrosion.

2. Centrifugal Pump
   Either open or closed impeller pumps are suitable if minimum clearance for granule passage is maintained. Speed of the pump should be in the range of 800–900 rpm to minimize degradation of the granules. A rubber or ceramic lined impeller is recommended for pump resistance to abrasion. Throttling of the discharge or suction should be avoided.

3. Eductor
   Water jet eductors have been used successfully in specialized situations where the other type mechanisms are not practical or available. They are easy to operate and require little maintenance.

4. Others

Slurries can also be transported using other types of pumps such as diaphragm and Moyno. Here, special problems may arise and the selection of a pump should be looked into carefully.

DEWATERING CARBON

Prior to thermal reactivation, excess water must be removed from the carbon before it is charged to the reactivation furnace. After the spent carbon has been thoroughly washed, the water-carbon slurry is hydraulically transported to a dewatering station above the furnace.
Slurries can be dewatered to low moisture content (50% wet basis) by drainage in a bin or tank. After the tank is filled with the carbon-water slurry, the initial rate of water drainage is very rapid. In most cases, 80% of the free water drains-out within 10 minutes. This constitutes the bulk of water in the voids between the granules. The balance of water on the outside of the granules consists of a film on the granule surface and webs between the granules, which drain quite slowly.

The rate of drainage is greater at higher water temperatures due to the reduction in surface tension and viscosity. Drainage time of 3½ hours with 180°F. water will provide quite free flowing carbon (less than 40% moisture, wet basis). Other techniques for continuous dewatering of granular carbon which have been used are:

1. Dewatering Screw
   Continuous dewatering screws have proven to be satisfactory for dewatering to a moisture content of 45-50%. The screw is fairly insensitive to fluctuations in feed conditions up to maximum capacity. For design information contact the nearest technical sales representative.

2. Drum Filters
   Top-feed vacuum drum filters have been used with reasonable success but are sensitive to feed conditions. If the carbon cake does not cover the entire screen cloth, the dewatering is not effective.

LOADING GRANULAR
CARBON COLUMNS
The recommended methods for hydraulic loading of granular carbon columns is to employ a blowcase, eductor or centrifugal slurry pump. When these methods are not practical, carbon can be loaded into the column in the dry state recommended procedures are followed.

HYDRAULIC LOADING PROCEDURES
One of the advantages of filling columns hydraulically is that the carbon can be wetted and partially de-aerated before it enters the column. This minimizes the possibility of air pockets in the column which lead to channeling. In moving bed systems, the reactivated carbon is sometimes transported to the column in treated process liquor. In fixed bed systems the use of a carbon-water slurry is usually the simplest and most economical means of moving carbon. Provisions should be made to maintain an adequate carbon-liquid ratio in the transport lines (1 pound carbon per gallon liquid).

When the column has been filled with slurry, there will be an excess of water. Therefore, the product discharge valve must be opened or an overflow arrangement made in order to discharge the excess liquid during the loading operation.

In a downflow system a minimum distance of 1 foot should be allowed from the top of the carbon bed to the tangent line of the vessel. The maximum distance between the top of the carbon bed and the tangent line is determined by the expected bed expansion during backwashing.

DRY LOADING PROCEDURES
To fill an empty column with granular carbon, the following procedure should be followed. Fill the column with hot water or pure process liquor to a depth of 2 feet. Empty bags of carbon into the liquid and at the same time introduce additional liquid into the column to maintain a supernatant layer 1 foot deep.

On smaller columns, an alternate procedure might prove easier. Fill a 25 gallon drum one-half full with liquid, then empty one bag of carbon into the drum. The carbon liquid slurry is then dumped into the 2 feet of liquid in the column. Additional liquid need not be added to the column during the filling operation when using this technique.

The liquid temperature should be maintained as high as possible when using the foregoing procedures. This promotes wetting and de-aeration of granular carbon.

BACKWASHING PROCEDURES
After the column has been filled with carbon, the bed should be backwashed by introducing hot water into the bottom of the column at a rate of ½-1 gpm/ft.² of bed area for one to two hours. This procedure will remove any fines which may have been created during handling, plus air pockets that may have been trapped in the bed. During backwashing, the flow rate should be controlled so that the carbon bed does not expand to the point where the particles classify (bed stratification).

DILUTING CHARACTERISTICS
Water will be displaced from the carbon as process liquor starts through a fixed bed which has been water loaded. Since this is not a true chromatographic type displacement, the process liquor will be diluted for a
FIGURE 8
DILUTION EFFECT OF COMING ON STREAM
IN A WATER LOADED GRANULAR CARBON COLUMN

short period of time after startup of a fresh bed. Figure 8 shows a typical dilution curve as a function of throughput for the start-up of a granular carbon system.

To minimize the amount of dilution, an attempt should be made to keep the heavier liquid below the lighter. If this is convenient and dilution still presents a problem, the flow rate should be reduced to 0.25 gpm/ft². When all the water has been displaced from the column, increase the flow to design rate.

If the heavier liquid is on top of the lighter (sugar syrup vs water) and excess dilution is undesirable, increasing the flow rate beyond design rate is sometimes helpful. At higher velocities, the higher density liquid tends to act like a hydraulic piston and pushes the lighter material ahead. Again, when the water has been displaced, return to design flow conditions.

CONSIDERATIONS OF WETTING CARBON WITH PROCESS LIQUOR

When the process liquor is not compatible with water or dilution is intolerable, it is recommended that the column loading procedure for water filling be followed, but treated process liquor should be substituted for water.

Several cautions should be exercised when using process liquor to wet the carbon. First, carbon-treated solution should be used to wet the carbon. This allows realization of the carbon's maximum purification power by having uncontaminated carbon available in the bol-
tom of the column, or in a down-stream position for a multiple column installation.

Second, if the product is heat sensitive, it may be necessary to "wet" the carbon first with a suitable solvent. This reduces heat generation caused by heats of adsorption and wetting and possible subsequent color formation.

Third, backwashing should be accomplished with hot carbon-treated solution.

**AFTER THE ADSORPTION CYCLE**

After granular activated carbon has become spent, the answers to several questions will determine the course to be followed.

1. Is the product adsorbed on the carbon or is the process liquor in the bed worth recovering?
2. If the process liquor is to be recovered, it must be displaced from the bed with a suitable liquid or air. At what point will the cost of recombining the process liquor override the value of the product?
3. Will the carbon be reactivated or discarded?
4. Can the carbon be reactivated by solution extraction, steam or will it require thermal regeneration in a reactivation furnace?

The following information is directed toward recovery of the process liquor and thermal reactivation of the spent carbon.
DISCHARGING THE COLUMN
In fixed bed systems the column may be emptied before or after washing. It can be discharged by gravity through a bottom outlet of 1-inch diameter or greater to a level below the column. If the slurry must be elevated to a point above the base of the column, pressure must be applied. Water or air pressure can be applied directly to the column (if design permits), or the slurry can flow by gravity to a blowcase or transport pump for elevation.

Dished or flat bottom columns are also employed for fixed bed systems. Removal of residual carbon-in the circumferential areas of the bottom of the column can be expedited by means of water injected above or below the support screen. Properly designed conical bottom columns do not require additional water to completely empty the column.

In moving bed systems the carbon column usually has a conical bottom similar to that shown in Figure 1. Slugs of spent carbon are periodically discharged from the column. The slug of spent carbon will flow through the carbon discharge valve by gravity at a concentration of approximately 3 pounds carbon/gallon of liquid. It is recommended that additional dilution of this stream be provided if long transport to the washing tank is required.

DIRECTION OF WASHING FLOW
In fixed bed systems, the spent carbon can be washed (removing residual product from carbon) “in situ” with water or a suitable solvent. Where moving bed systems are used, the spent carbon is transported to a washing tank.

The direction of washing, downflow or upflow, will depend on the relative specific gravity of solvent and process liquor for minimum dilution. If the process liquor has a lower specific gravity than the solvent, upflow washing should be used. The system plotted in Figure 9 had a process liquor with a higher specific gravity and consequently, the downflow system created less dilute solution for recovery of more process liquor. The proper water or solvent temperature will depend on the individual application, but should be as high as possible without excessive removal of the impurities from the carbon.

RECOMMENDED FLOW RATES
To recover process liquor, a wash rate of 0.50 gpm/ft.² is generally recommended. With this procedure, the volume of diluted liquor recovered should be 1 ½ to 2 times the volume of the carbon. A variation of this procedure can be used to minimize the amount of dilute solution obtained, if this is a problem. This technique involves a solvent flow rate of 0.25 gpm/ft.² until product concentration in the effluent is zero or the lowest practical level for recovery.

REACTIVATION PREPARATION
To prepare carbon for furnace reactivation, it should be washed for a minimum of two hours at a flow rate of 1 gpm/ft.² or higher in addition to any previous washing for product recovery. Hot water will remove some adsorbed materials, but more importantly it should wash inorganics from the carbon. Washing efficiency is enhanced in some applications by prewashing with one volume of dilute 1-3% caustic (NaOH) or acid (HCl).

After washing, the carbon can be sluiced to the dewatering station for subsequent feeding to the furnace.
CAUTION!

1. Oxygen Demand Created by Activated Carbon in Confined Vessels

It has been confirmed that wet granular activated carbon confined in large vessels creates an oxygen demand which is hazardous to human health and can cause death unless proper safety precautions are observed. Investigation of this matter was prompted by an accident which occurred on a project in which a granular activated carbon system was being installed.

Studies conducted in vessels similar to that in which the accident occurred have shown that low oxygen content exists in vessels containing wet carbon. Laboratory experiments conducted since that time also have revealed that commercial activated carbons in a wet or moist condition—including Filtrasorb and Pittsburgh grades—will lower the oxygen content of an isolated space. Preliminary indications of this research are:

a. The phenomenon occurs with wet activated carbon of all common types.
b. The rate of oxygen uptake naturally varies with the degree of exposure of the wet carbon to the air. Thus it is relatively rapid in a drained bed.
c. There is some indication of a limit to carbon's capacity for oxygen, but until more is known, it would be prudent to assume that all carbon (fresh, used, reactivated) will also exhibit this characteristic. Similarly, although these tests were run with water, it should be assumed that the phenomenon will occur in other liquid and vapor systems.

All confined spaces, including those containing activated carbon, should be presumed to be hazardous. Appropriate safety measures should always be taken before entering, as well as when workers are in, a confined space. OSHA regulations applicable to respiratory protection in oxygen-deficient atmospheres should be strictly adhered to.

2. Hazards with Certain Process Conditions

Under certain process conditions, activated carbons may show an affinity for atmospheric oxygen or may interact with process streams to generate potentially toxic or hazardous levels of hydrogen sulfide, methane, ethanol, carbon dioxide and other gases. These effects can become pronounced in a relatively confined space, such as the headspace of an adsorber. Should entry to confined spaces containing activated carbon become necessary, appropriate ventilation and other safety practices for potentially flammable, toxic or oxygen-deficient environments should be followed.

To avoid possible combustion of the carbon or the material being adsorbed, caution is recommended in contacting activated carbon with strong oxidizing agents, such as chlorine. Use with liquid oxygen and ozone is discouraged.

3. Need for Grounding of Carbon Systems

In certain systems, high voltage static electrical charges may accumulate to levels of shock or ignition hazard. As a precaution against possible ignition or shock, all carbon treatment systems should be adequately grounded.
Appendix H-2
Carbon Column Loading With an Eductor
**Fig. 28. TYPE 254 HOPPER-EQUIPPED EDUCTOR.** Standard units are made in cast iron and have hardened steel nozzles and throat bushings. Special materials are also available on special order. Both nozzles and throat pieces are removable and pressure and discharge connections can be fitted for ease where maximum portability is desired. Handles and supporting feet are optional.

*Fig. 29. TYPE 254 EDUCTOR.*

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**TABLE 7. SIZES AND DIMENSIONS OF SK HOPPER-EQUIPPED EDUCTORS, TYPE 254.**

<table>
<thead>
<tr>
<th>Size in Inches</th>
<th>Connections in Inches</th>
<th>Dimensions in Inches</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Discharge</td>
<td>Pressure</td>
</tr>
<tr>
<td>1 1/2</td>
<td>1 1/2</td>
<td>1 1/4</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>3</td>
<td>3</td>
<td>2 1/2</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
<td>4</td>
</tr>
</tbody>
</table>

**Type 254 Eductor** is made for handling slurries or solids in granular form and are used extensively for ejecting sludges from tank bottoms, for pumping sand from filter beds and for washing and conveying granular materials. Typical materials handled include: bauxite, charcoal, diatomaceous earth, lime, marl, fly ash, rosin, rock and granulated salt, sawdust, caustic soda ash, dry sodium nitrate, powdered sulphur, wheat and many others.

A typical application of a Fig. 254 Eductor is shown in the application section.

Agitating jets keep the material from adhering to the sides of the hopper and keep it moving down into the eductor. Pressure water, passing through the eductor nozzle, entrains the sand, sludge or other materials and discharges into the piping system. Under normal operating conditions, the pressure should be 4 times the discharge head and the eductor should handle from 1 to 3 lbs. of sand per gallon water. It will handle equivalent volumes of other materials. For performance information, see accompanying Technical Data.
Appendix I

Water Quality Testing
Appendix I-1

PBA Industrial Waste Water Permit (NPDES)
### A-5 EFFLUENT LIMITATIONS AND MONITORING REQUIREMENTS

During the period beginning July 1, 1984 and lasting through the date of expiration Aug 16, 1996, the permittee is authorized to discharge from outfall(s) serial number(s) 002, combined industrial effluents. Such discharges shall be limited and monitored by the permittee as specified below:

<table>
<thead>
<tr>
<th>Effluent Characteristic</th>
<th>Discharge Limitations</th>
<th>Other Units (Specify)</th>
<th>Monitoring Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>kg/day (lbs/day)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Daily Avg</td>
<td>Daily Max</td>
<td>Daily Avg</td>
</tr>
<tr>
<td>Flow—m³/Day (MGD)</td>
<td>N/A</td>
<td>N/A</td>
<td>0.3</td>
</tr>
<tr>
<td>Total Organic Carbon</td>
<td>17 (38)</td>
<td>45 (100)</td>
<td>15 mg/l</td>
</tr>
<tr>
<td>Total Suspended Solids</td>
<td>74.5 (164)</td>
<td>111 (245)</td>
<td>N/A</td>
</tr>
<tr>
<td>Oil &amp; Grease</td>
<td>24.8 (54.6)</td>
<td>37.2 (81.8)</td>
<td>N/A</td>
</tr>
<tr>
<td>Phosphate (total) as P</td>
<td>5.0 (10.9)</td>
<td>9.9 (21.8)</td>
<td>N/A</td>
</tr>
<tr>
<td>Phosphate (ortho) as P</td>
<td>2.5 (5.5)</td>
<td>5.0 (10.9)</td>
<td>N/A</td>
</tr>
<tr>
<td>Barium</td>
<td>5.0 (10.9)</td>
<td>9.9 (21.8)</td>
<td>N/A</td>
</tr>
<tr>
<td>Cadmium, Total</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Lead, Total</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Zinc, Total</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Temperature</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Dissolved Oxygen</td>
<td>N/A</td>
<td>N/A</td>
<td>5.0 mg/l</td>
</tr>
</tbody>
</table>

*Report
**Minimum value

See Part III, Paragraphs A & B

The pH shall not be less than 6.0 standard units nor greater than 9.0 standard units and shall be monitored by continuous record.

There shall be no discharge of floating solids or visible foam in other than trace amounts.

Samples taken in compliance with the monitoring requirements specified above shall be taken at the following location(s):

At the outfall from the industrial waste treatment plant.
Appendix I-2

Results of Chronic Toxicity Testing of Effluent After Carbon Treatment on Daphnia Magna
Appendix I-2

Results of the Chronic Toxicity Test of Effluent After Carbon Treatment on *Daphnia Magna*

<table>
<thead>
<tr>
<th>Day Number</th>
<th>Control 6.25</th>
<th>12.5</th>
<th>25.0</th>
<th>50.0</th>
<th>100.0</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Percent of Control</td>
<td>Percent of Control</td>
<td>Percent of Control</td>
<td>Percent of Control</td>
<td>Percent of Control</td>
</tr>
<tr>
<td>Day 7</td>
<td>96.7 100.0</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
</tr>
<tr>
<td>Percent Survival (Rep 1-3)</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
</tr>
<tr>
<td>Total Young (Rep 4-10)</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
</tr>
<tr>
<td>Total Young/Adult (Rep 4-10)</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
<td>0 0</td>
</tr>
<tr>
<td>Total Young/Adult/Day (Rep 4-10)</td>
<td>0.14 0.59</td>
<td>0.64 0.58</td>
<td>0.93 1.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Day 14</td>
<td>96.7 100.0</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
</tr>
<tr>
<td>Percent Survival (Rep 1-3)</td>
<td>28 58</td>
<td>63 57</td>
<td>78 186</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total Young (Rep 4-10)</td>
<td>2 8.3</td>
<td>415 450</td>
<td>8.1 405</td>
<td>13 650</td>
<td>26.6 5320</td>
</tr>
<tr>
<td>Total Young/Adult (Rep 4-10)</td>
<td>0.14 0.59</td>
<td>0.64 0.58</td>
<td>0.93 1.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total Young/Adult/Day (Rep 4-10)</td>
<td>0.40 1.02</td>
<td>1.50 1.58</td>
<td>2.80 4.69</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Day 21</td>
<td>86.7 93.3</td>
<td>100.0</td>
<td>93.3</td>
<td>93.3</td>
<td>93.3</td>
</tr>
<tr>
<td>Percent Survival (Rep 1-3)</td>
<td>119 128</td>
<td>220 199</td>
<td>353 690</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total Young (Rep 4-10)</td>
<td>8.5 21.3</td>
<td>251 370</td>
<td>33.2 391</td>
<td>58.8 692</td>
<td>98.6 1160</td>
</tr>
<tr>
<td>Total Young/Adult (Rep 4-10)</td>
<td>0.40 1.02</td>
<td>1.50 1.58</td>
<td>2.80 4.69</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Appendix J
Carbon Loading and Regeneration Computations
Appendix J-1

CARBON CONSUMPTION

Carbon consumption depends on three factors:

1. Quantity of wastewater treated.
2. Concentration of waste compounds in the water which adsorb onto one pound of carbon.

QUANTITY OF WASTEWATER

The mean daily flow rate for CWTF effluent for the six-month period Dec 1981 - May 1982 was reported as 238,369 GPD, with a standard deviation of 129,512 GPD and 95% statistical maximum of 497,393 GPD. More recent data for May 1983 - April 1984 yield a mean of 245,042 GPD, standard deviation of 103,665 GPD and 95% statistical maximum of 452,371 GPD. Based on these data, a design value of 250,000 GPD is taken for normal, current flow rate. An allowance for excursions up to 500,000 GPD will be considered as part of normal operation. Mobilization rates are taken as 650,000 GPD.

CONCENTRATION OF WASTE

The primary measurement for waste amenable to carbon treatment has been Total Organic Carbon (TOC). For the period Dec 1981 - May 1982, mean TOC in CWTF feed was 7.4 ppm with a 95% statistical max of 15.6 ppm. More recent data has had a rangeability of 10 to 30 ppm. Analyses for specific pollutants like hexachloroethane (HEX) is not available but is included as a part of the TOC analysis. EPA limits for TOC are 15 ppm daily average and 40 ppm absolute maximum. However, since HEX is on the RCRA hazardous list, individual limits may be established for it in the future. For carbon loading computations, total removal of 30 ppm TOC is assumed. It is recognized that substituents not measured by TOC may adsorb and form part of the adsorbate load.

CARBON LOADING

Preliminary adsorption isotherm tests on PBA waste indicate a 2g adsorbate per gram of carbon loading. This seems quite high compared to literature values and a design value of 0.5 g/g is more typical. A third figure is derived from data for powdered carbon use at the CWTF. Based on an average of 400 lb/day of carbon, 250,000 GPD rate and 30 ppm TOC removal, a loading of 0.156 g/g is computed.
Carbon consumption for the three loading assumptions and two flow rates (normal 250,000 GPD and mobilization 650,000 GPD) assuming 30 ppm TOC removal. Carbon consumption at the 0.156 g/g level is typical of the powdered carbon system. Due to insufficient contact time and single stage slurry operation this loading is expected to be low as compared to loading achieved with a granular fixed bed unit.

<table>
<thead>
<tr>
<th>FLOW RATE (GPD)</th>
<th>LOADING (2) g ADSORBATE/g CARBON</th>
<th>CARBON CONSUMPTION (1) lb/day</th>
<th>lb/Year</th>
<th>ft³/Year</th>
</tr>
</thead>
<tbody>
<tr>
<td>250,000</td>
<td>2.0</td>
<td>31.24</td>
<td>11,402</td>
<td>380</td>
</tr>
<tr>
<td>250,000</td>
<td>0.5</td>
<td>124.95</td>
<td>45,607</td>
<td>1,520</td>
</tr>
<tr>
<td>250,000</td>
<td>0.156</td>
<td>400.00</td>
<td>146,175</td>
<td>4,873</td>
</tr>
<tr>
<td>650,000</td>
<td>2.0</td>
<td>81.22</td>
<td>29,645</td>
<td>988</td>
</tr>
<tr>
<td>650,000</td>
<td>0.5</td>
<td>324.87</td>
<td>118,578</td>
<td>3,953</td>
</tr>
<tr>
<td>650,000</td>
<td>0.156</td>
<td>1,040.00</td>
<td>380,055</td>
<td>12,669</td>
</tr>
</tbody>
</table>

(1) Based on removal of 30 ppm TOC with TOC as only adsorbate
(2) Three loading levels based on the following:
   (a) 2.0 g/g is from preliminary isotherm tests
   (b) 0.5 g/g is a typical literature value
   (c) 0.156 g/g is based on powdered carbon rate in existing facility.
Appendix J-3

GRANULAR CARBON FIXED BED ADSORBER SYSTEM SIZING

Two design criteria were used in the following system sizing:

1. Hydraulic rate of 2 - 4 GPM/FT$^2$ of flow area.

2. Total retention time of 30 - 60 minutes, calculated as void volume in bed (50% of bed volume) / volumetric flow rate.

Two system designs were considered, a single train and a dual parallel train system. Description of these are as follows:

**Single Train System**

2 Adsorbers in series, 12' diameter x 14' height of bed.

**Dual Train System**

4 adsorbers, 10' diameter x 10' height of bed

2 parallel trains

2 adsorbers in series in each train

Hydraulic rates and retention times are given for the above two systems for a variety of flow rates in Appendix J-4. The flow rates used represent current average (250,000 GPD), current typical maximum (500,000 GPD) and mobilization (650,000 GPD).

The concept of two columns in series gives a certain safety margin in the system operation. When the capacity of the first column in a train is exhausted, most of the second column capacity should still be intact. The first column can be blocked out while flow continues through the second while the first column is emptied and recharged with fresh carbon. The two column concept also provides extra capacity for high flow rate or high concentration excursions.

The 2-4 CPM/FT$^2$ design criteria is based on downflow adsorbers. Calgon recommends these as opposed to upflow adsorbers. If upflow adsorbers can be used then flows of 5-7 GPM/FT$^2$ could be used. This would result in smaller diameter but taller adsorbers.
Appendix J-4

GRANULAR CARBON ADSORBER SYSTEM SIZING (PROPOSED SYSTEM)

<table>
<thead>
<tr>
<th>System</th>
<th>Flow (5) GPD</th>
<th>Hydraulic Rate (3) GPM/FT²</th>
<th>Retention Time, Min (4)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dual Train (1)</td>
<td>250,000</td>
<td>1.105</td>
<td>67.7</td>
</tr>
<tr>
<td>Dual Train</td>
<td>500,000</td>
<td>2.210</td>
<td>33.8</td>
</tr>
<tr>
<td>Dual Train</td>
<td>650,000</td>
<td>2.837</td>
<td>26.0</td>
</tr>
<tr>
<td>Single Train (2)</td>
<td>250,000</td>
<td>1.535</td>
<td>68.2</td>
</tr>
<tr>
<td>Single Train</td>
<td>500,000</td>
<td>3.070</td>
<td>34.1</td>
</tr>
<tr>
<td>Single Train</td>
<td>650,000</td>
<td>3.991</td>
<td>26.2</td>
</tr>
</tbody>
</table>

(1) Dual train system consists of 2 parallel trains, each with 2 adsorbers in series, each adsorber 10' dia X 10' bed ht.

(2) Single train system consists of 2 adsorbers in series, each adsorber 12' dia X 14' bed ht.

(3) Typical design values are 2-4 GPM/FT²

(4) Typical retention times are 30-60 min.

(5) The three flow rates shown represent current average rates (250,000 GPD), current typical maximum rates (500,000 GPD), and mobilization rates (500,000 GPD).
Appendix J-5

POWDERED CARBON SYSTEM OPERATING COSTS ($/yr) FOR EXISTING SYSTEM

<table>
<thead>
<tr>
<th></th>
<th>250,000 GPD</th>
<th>650,000 GPD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon Cost (1)</td>
<td>48,238</td>
<td>125,418</td>
</tr>
<tr>
<td>Labor, Maintenance (2)</td>
<td>33,596</td>
<td>67,192</td>
</tr>
<tr>
<td>Landfill Disposal Cost (3)</td>
<td>22,790</td>
<td>59,254</td>
</tr>
<tr>
<td>Sludge Handling Cost (4)</td>
<td>10,000</td>
<td>20,000</td>
</tr>
<tr>
<td><strong>TOTAL:</strong></td>
<td><strong>$114,624</strong></td>
<td><strong>$271,864</strong></td>
</tr>
</tbody>
</table>

(1) Carbon cost based on historical use rate of 400 lb/day at 250,000 GPD, $.33/lb. For mobilization rate, usage assumed proportional to flow rate.

(2) Based on information from Charles Carroll, Foreman, CWTF. Carbon system requires attention of one man from 1/4 to full time, depending on quantity of dye coming through system. 2 shifts per day, Mon-Fri., 1 shift per day on weekends. Take as an average 1/2 man per shift (i.e. 4 hours per shift) at $13.96/hr. Assume labor doubles for mobilization rates.

(3) Based on $90.00/yd^3 to construct a RCRA landfill. Sludge volume due to carbon taken as equal to dry carbon volume.

(4) Estimated cost to remove sludge from settling lagoon and transport to landfill.
Appendix J-6

COMPARISON OF OPTION 2 (GRANULAR CARBON - CONTRACT REGENERATION) VS. OPTION 1 (POWDERED CARBON - LANDFILL DISPOSAL)

Operating costs for Option 2 (Granular Carbon - Contract Regeneration) are given below. Costs are shown for two levels of carbon loading (0.5 g/g typical from literature and 1.0 g/g more optimistic value based on preliminary lab data).

A comparison of these operating costs for Option 2 vs the existing treatment Option 1 (Powdered Carbon - Landfill Disposal) is given in Appendix J-7. The operating cost savings is shown and the payout computed based on a cost estimate of $450,000 to install granular carbon adsorbers. The table shows that payout is 5-7 years at current average rates, 2-3 years at mobilization rates.

**GRANULAR CARBON OPERATING COST (PROPOSED SYSTEM)**

<table>
<thead>
<tr>
<th>Flow Rate, GPD</th>
<th>250,000</th>
<th>250,000</th>
<th>650,000</th>
<th>650,000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon Loading, g/g carbon (1)</td>
<td>0.5</td>
<td>1.0</td>
<td>0.5</td>
<td>1.0</td>
</tr>
<tr>
<td>Carbon Cost, $/yr (2)</td>
<td>45,600</td>
<td>22,800</td>
<td>118,600</td>
<td>59,300</td>
</tr>
<tr>
<td>Labor Maintenance $/yr (3)</td>
<td>5,000</td>
<td>5,000</td>
<td>10,000</td>
<td>10,000</td>
</tr>
<tr>
<td>TOTAL OPERATING COST</td>
<td>$50,600</td>
<td>$27,800</td>
<td>$128,600</td>
<td>$69,300</td>
</tr>
</tbody>
</table>

(1) Adsorbance capacity of carbon, g waste adsorbed/g carbon

(2) Assumes contract regeneration service. Cost of $1.00/lb covers cost to regenerate and transport carbon in contractor's trucks.

(3) Based on minimal attention required for fixed bed adsorbers, 1/2 man-hour/shift at $13.96, 2 shifts/day Mon-Fri., 1 shift per day on weekends. Assume labor cost doubles at mobilization rates.
### COMPARISON OF OPTION 1 (POWRED CARBON LANDFILL DISPOSAL) VS OPTION 2 (GRANULAR CARBON - CONTRACT REGENERATION)

<table>
<thead>
<tr>
<th>Rate, GPD</th>
<th>Granular Carbon Loading, g/g</th>
<th>Option 1 Cost $/yr (1)</th>
<th>Option 2 Cost $/yr (2)</th>
<th>Option 2 Savings $/yr (3)</th>
<th>Payout, Years (4)</th>
</tr>
</thead>
<tbody>
<tr>
<td>250,000</td>
<td>0.5</td>
<td>114,624</td>
<td>50,600</td>
<td>64,024</td>
<td>7.0</td>
</tr>
<tr>
<td>250,000</td>
<td>1.0</td>
<td>114,624</td>
<td>27,800</td>
<td>86,828</td>
<td>5.2</td>
</tr>
<tr>
<td>650,000</td>
<td>0.5</td>
<td>271,864</td>
<td>128,600</td>
<td>143,246</td>
<td>3.1</td>
</tr>
<tr>
<td>650,000</td>
<td>1.0</td>
<td>271,864</td>
<td>69,300</td>
<td>202,564</td>
<td>2.2</td>
</tr>
</tbody>
</table>

(1) From Appendix J-5
(2) From Appendix J-6
(3) Difference between Option 1 cost and Option 2 cost.
(4) Payout for $450,000 investment to install granular carbon fixed bed adsorption system, computed as $450,000 divided by Option 2 Savings ($/yr) = Payout (years).
Appendix J-8

COMPARISON OF OPTION 3 (GRANULAR CARBON - CONTRACT REGENERATION) VS. OPTION 2 (GRANULAR CARBON - PBA REGENERATION)

A study of the cost effectiveness of self-owned and operated regeneration vs contract regeneration was made. Results are presented in Appendix J-9. Option 2 (Contract Regeneration) costs are taken at $1.00/lb of carbon consumed, which includes cost to transport spent carbon to contract regenerator in his truck, regenerate carbon and transport carbon back to PBA. Since virgin carbon also costs about $1.00/lb, make-up due to attrition and regeneration losses is also included at $1.00/lb, of carbon consumed.

Option 3 costs are based on data from a vendor of carbon regeneration systems (Shirco). Data from Shirco for their smallest unit (as 100 lb/hr capacity) indicates operating costs are 21 cents/lb. Due to the high HCl content, an additional ten cents/lb was allowed to cover increased maintenance costs. The Shirco unit is an electrical furnace and afterburner made of steel with an internal insulation blanket of silica/alumina. High HCl content resulting from hexachloroethane (HEX) will result in corrosion to the unit. Shirco indicates that other customers treat such corrosion as a maintenance problem, inspecting and repairing the unit as needed. Ten percent losses are assumed for combined attrition and regeneration losses, which adds another ten cents/lb to the cost, based on total carbon consumed. Investment for the unit is $400,000 plus an estimated $200,000 to provide utilities and site preparation. Shirco's cost of $400,000 includes all pumps, vessels, furnace, afterburner, scrubber and controls. Costs are summarized below:

SHIRCO INCINERATION REGENERATION SYSTEM COSTS

<table>
<thead>
<tr>
<th>Operating Costs</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Utilities, Labor, Maintenance</td>
<td>$0.21/lb</td>
</tr>
<tr>
<td>Extra Maintenance due to hi HCL</td>
<td>$0.10/lb</td>
</tr>
<tr>
<td>Carbon, 10% loss at $1.00/lb</td>
<td>$0.10/lb</td>
</tr>
<tr>
<td><strong>TOTAL</strong></td>
<td><strong>$0.41/lb</strong></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Investment</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Shirco Unit (100 lb/hr)</td>
<td>$400,000</td>
</tr>
<tr>
<td>Utilities, Site Preparation</td>
<td>$200,000</td>
</tr>
<tr>
<td><strong>TOTAL</strong></td>
<td><strong>$600,000</strong></td>
</tr>
</tbody>
</table>
## Appendix J-9

COST COMPARISON OF OPTION 3 (GRANULAR CARBON - PBA REGENERATION) VS OPTION 2 (GRANULAR CARBON - CONTRACT REGENERATION)

<table>
<thead>
<tr>
<th>Flow Rate GPD</th>
<th>Granular Carbon Loading, g/g</th>
<th>Option 2 Cost $/yr</th>
<th>Option 3 Cost $/yr</th>
<th>Option 3 Savings $/yr</th>
<th>Payout, Years</th>
</tr>
</thead>
<tbody>
<tr>
<td>250,000</td>
<td>0.5</td>
<td>45,600</td>
<td>18,696</td>
<td>26,904</td>
<td>16.7</td>
</tr>
<tr>
<td>250,000</td>
<td>1.0</td>
<td>22,800</td>
<td>9,348</td>
<td>13,452</td>
<td>33.5</td>
</tr>
<tr>
<td>650,000</td>
<td>0.5</td>
<td>118,600</td>
<td>48,626</td>
<td>69,974</td>
<td>6.4</td>
</tr>
<tr>
<td>650,000</td>
<td>1.0</td>
<td>59,300</td>
<td>24,313</td>
<td>34,987</td>
<td>12.9</td>
</tr>
</tbody>
</table>
Appendix J-10
Carbon Regeneration by Incineration (Shirco, INC) at PBA
SHIRCO CARBON
REGENERATION SYSTEMS
SHIRCO CARBON REGENERATION SYSTEMS

Advantages of In-site Regeneration
- Process control
- Quality of carbon assured
- Control of trace elements in carbon
- Return on investment as opposed to continuing outside fees

Features of System
- Precise process control
- Gentle carbon handling
- Efficient energy usage
- Low emissions
- Pre-assembled/skid-mounted
- Easy access for maintenance
- Readily integrated into existing facility
- Commercially available spare parts
### STANDARD REGENERATION SYSTEMS

<table>
<thead>
<tr>
<th>Model # (Granular)</th>
<th>Model # (200 CRS)</th>
<th>Model # (400 CRS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 CRS</td>
<td>200 CRS</td>
<td>400 CRS</td>
</tr>
<tr>
<td><strong>$</strong></td>
<td><strong>$</strong></td>
<td><strong>$</strong></td>
</tr>
<tr>
<td><strong>Approximate Size</strong></td>
<td><strong>$</strong></td>
<td><strong>$</strong></td>
</tr>
<tr>
<td>4' x 23'</td>
<td>5' x 26'</td>
<td>6' x 35'</td>
</tr>
<tr>
<td><strong>Regenerated Output (Granular)</strong></td>
<td><strong>$</strong></td>
<td><strong>$</strong></td>
</tr>
<tr>
<td>100 lbs./hr.</td>
<td>200 lbs./hr.</td>
<td>400 lbs./hr.</td>
</tr>
<tr>
<td><strong>Approximate Cost per Pound</strong></td>
<td><strong>$</strong></td>
<td><strong>$</strong></td>
</tr>
<tr>
<td>21¢</td>
<td>16¢</td>
<td>12¢</td>
</tr>
</tbody>
</table>

ENGINEERED REGENERATION SYSTEMS ALSO AVAILABLE.
Figures computed on operation 24 hours per day, 5 days per week, 50 weeks per year.
Table is based on historical experience but is intended for illustration only.

**Applications**
- Drinking water systems
- Wastewater treatment systems
- Food and beverage processing industry
- Chemical industry
- Vapor phase

**System includes:**
- Infrared regenerator
- Dewatering feed screw
- Afterburner
- Venturi scrubber
- Exhaust gas ducting and stack
- Power control centers
- Afterburner combustion blower
- Induced draft blower
- Control panel
- Quench tank

Shirco, Inc.
1195 Empire Central
Dallas, Texas 75247
(214) 630-7511
SHIRCO COMPANY PROFILE

Shirco, Inc., a leading manufacturer of infrared thermal process systems, is based in Dallas, Texas. Since its inception in the mid-1970's, the company has seen steady growth and its products have become widely accepted throughout the United States and the world. Shirco, an industry leader in thermal processing, has built a reputation of excellence in custom-engineered systems featuring patented infrared technology as well as conventional systems. As an innovative multiple-application thermal process company, Shirco offers both standard and engineered products for industrial and municipal applications.

Typical Applications of Shirco Systems:
- Carbon Regeneration
- Hazardous Waste Incineration
- Municipal Sludge Incineration
- Industrial Sludge Incineration
- Fume Incineration
- Liquid Incineration
- Flare Systems
- Special Thermal Process Applications

Shirco Support Services:
- Pilot Test Facility –
- U.S. EPA "RCRA" Permit in Effect
- Analytical Services
- Laboratory Services
- Process Development
- Systems Responsibility
- Field Installation and Maintenance
- Construction and Operating Permit Assistance

For information, contact our home office:
(Shirco, Inc)
195 Empire Central
Dallas, Texas 75247
(214) 630-7511

Or contact our local representative in your area:
PILOT FACILITY AND ANALYTICAL CAPABILITIES
Shirco has a fully-equipped laboratory and pilot facility in Dallas. The laboratory can perform a full range of property analysis and bench-scale thermal processes. The pilot furnace can function as an incinerator, regeneration or activation furnace, or dryer in addition to a variety of specialty process configurations. High-temperature afterburning and pollution abatement equipment are incorporated in the system. The pilot facility and laboratory both have "existing facility" status under the recent EPA Hazardous Waste Management Regulations. As such, they are available for analysis and trial burns on hazardous wastes.

ANALYSIS AND CONSULTATION
Shirco engineers can assist in defining your thermal processing requirements and selecting the right equipment for your needs. Our expertise in environmental regulations and permits is available to help you in obtaining facility certification under state and federal regulations.

FIELD SERVICE
Shirco's Field Service Group is experienced in the installation, start-up, and maintenance of thermal processing equipment. Technical assistance is readily available upon request. Customer operating personnel will be thoroughly trained on Shirco equipment to insure proper operation and maintenance. Complete Operation and Maintenance Manuals are provided for all equipment.

PROCESS DEVELOPMENT
Shirco can assist in determining the feasibility of your new process. Our Process Development Department can provide a total development package from preliminary analysis through pilot testing. Work by Shirco can be done under Secrecy Agreements with your firm to protect your proprietary process information.

SHIRCO THERMAL SYSTEMS
- Activated Carbon Regeneration
- Hazardous Waste Disposal
- Industrial Liquid Waste Incineration
- Process Furnaces
- Fume Incineration
- Sulfur Reaction Furnaces
- Elevated and Ground Level Flares
- Sludge Drying and Incineration

Shirco, Inc
Representatives in major U.S. cities and in Europe and Japan
Executive Offices and Manufacturing Plant Located At
1195 Empire Central • Dallas, Texas 75247 • (AC 214) 630-7511
ACTIVATED CARBON REGENERATION

Shirco process systems have been used for many years in the economical and efficient regeneration of activated carbon. Both powdered and granular carbons have been regenerated from a wide range of industrial applications, including food processing, waste water treatment, potable water treatment, and vapor recovery.

Activated carbon is an extremely effective means of treating many process streams, but its use is frequently dependent upon economical regeneration. Shirco carbon systems offer the highest available degree of control of the critical variables of residence time, atmosphere and temperature profile. This results in the best recovery of carbon properties with the lowest possible losses. The range of economical carbon regeneration with Shirco equipment can start as small as less than 100 pounds per hour. Single units are available for requirements up to as large as 35 million pounds per year.

Shirco systems are in service with several types of activated carbon. Powdered carbon, PACT sludge and granular carbon can be regenerated with the Shirco process. Based on this experience, Shirco can assist the customer in selection of the optimum activated carbon process. Each Shirco system is carefully engineered to be the most effective solution to the customer’s regeneration requirements.

SOLID WASTE AND SLUDGE DISPOSAL

Shirco systems offer process versatility and can incinerate a wide range of solid and semi-solid materials. Efficient design and energy recovery are employed to provide each customer with a cost-effective solution to the specific waste disposal problem.

Shirco has been a pioneer in the application of infrared technology to the difficult problems of sludge incineration. Both gas and electric energy can be employed, as required, as auxiliary energy to control the efficiency of the incineration process. Shirco uses the most recent developments in insulation materials, process controls and instrumentation, waste heat recovery, and air pollution control equipment to provide the most economical incinerator for each application.

MULTIPROCESS FURNACES

A unique feature of Shirco furnaces is that they can be designed to efficiently perform multiple processes in the same system. One installed Shirco system regenerates activated carbon, and then can be quickly and automatically converted to incinerate sludge. Systems that combine various processes or various materials — such as liquid and solids — in one Shirco unit can reduce both overall capital investment and plant operating cost.
Appendix J-11

COMPARISON OF OPTION 4 (GRANULAR CARBON - LANDFILL DISPOSAL) VS. OPTION 2 (GRANULAR CARBON - CONTRACT REGENERATION)

Another option considered is to use granular carbon but not bother with regeneration since volumes are so small, but merely landfill the spent carbon. Since Calgon's quotes indicate $1.00/lb approximately for either virgin or regenerated carbon, the extra cost for landfill (11.1 cents/lb based on $90/yd$^3$ of carbon disposed) would make Option 4 more costly. However, if some alternate carbon supplier would turn out to be less expensive than Calgon, say 80 cents/lb for virgin carbon, then costs would favor the landfill option. This is not likely and the concept of stockpiling toxic waste at PBA, even in a RCRA landfill, doesn't seem to make sense when a final disposition to harmless by-products via regeneration is possible.
Appendix J-12

Carbon Regeneration by Contractor Service (Calgon Corp)
A unique wastewater treatment service offered by Calgon Corporation makes available to industrial plants the benefits of the granular activated carbon process for the physical/chemical removal of organic pollutants without major capital investment.
This shop-fabricated system can be placed on line within a few hours after reaching plant site.

Fresh supply of reactivated carbon is quickly unloaded at plant site from a specially designed Calgon bulk hopper truck.
Calgon Corporation offers a water pollution control service on your plant site to relieve you of the problem of treating dissolved organic wastes. The Calgon Carbon Service combines the proven reliability of the carbon adsorption process with the economies of carbon reactivation and modular shop-fabricated equipment. Systems based on the use of standard equipment modules are tailored to meet your specific dissolved organic problem. The equipment can be rapidly installed at your plant location. This unique approach to industrial water pollution control greatly reduces the time, complexity and cost of installing an activated carbon adsorption system. Full cost of the Calgon Carbon Service is covered by a monthly fee which is determined by thorough testing prior to the installation of the equipment. The fee is usually 15-20% less than the annualized operating expense for a system that you would design, procure, install, operate and maintain yourself.

Major features and benefits of the Calgon Carbon Service:
1. It requires no major capital investment—your capital is reserved to use for your own revenue-generating alternatives.
2. The system can be installed rapidly, with minimum disruption of plant operations.
3. It produces high quality effluent. In many cases, this treated wastewater is of such a quality that it can be recycled.
4. Trained personnel of the Calgon Activated Carbon Division maintain the equipment, make available monitoring and analytical work, issue periodic reports on results obtained and, if desired by the client, can assume responsibility for operation of the adsorption system.
5. It is adaptable to future, possibly much more stringent, stream standards.
6. Its effectiveness for any given application can be demonstrated conclusively prior to installation.
7. Calgon assumes responsibility for carbon supply, handling and reactivation.
8. It eliminates the concern about air-pollution problems, since Calgon reactivates the carbon at one of its central reactivation facilities.
10. It provides an effective and economical solution to dissolved organic pollution.
11. It provides ultimate organic pollution disposal via total destruction.
12. It eliminates concerns and long-term liabilities associated with other methods of organic pollution disposal.

*Calgon Carbon Service is a service mark of Calgon Corporation.*
This is just one of many adsorption systems Calgon has installed at industrial plants throughout the United States and Europe.

Most effective technology. Adsorption with granular activated carbon has been proven to be the most cost/effective means of removing toxic and non-toxic dissolved organic pollutants from wastewater.

Unlike other treatment systems, granular carbon systems are immune to attack by toxic substances and are less affected by changes in wastewater temperature, pH or mixtures of organic pollutants.

When used to remove toxic and hazardous organic substances, carbon adsorption can upgrade existing facilities by removing such contaminants at the point source. Thus carbon adsorption can be applied prior to biological treatment systems effectively and economically.

Calgon has evaluated effectiveness of carbon adsorption on hundreds of complex wastewaters from many industrial plants. These tests have demonstrated that granular carbon is highly effective in treating most organic wastes and mixtures of organic wastes.

In general, compounds which are non-polar and of higher molecular weight (in the broad range of from four to 20 carbon atoms) can be quickly and easily removed from wastewater by activated carbon. The following classes of compounds are readily adsorbed on carbon:

- Aromatic Hydrocarbons (benzene, toluene, xylene)
- Polynuclear Aromatics (naphthalene, anthracene, biphenyls)
- Chlorinated Aromatics (chlorobenzene, polychlorinated biphenyls, aldrin, endrin, toxaphene, DDT)
- Phenolics (phenol, cresol, resorcinol and polyphenyls such as tannin and lignin derivatives)
- Chlorinated Phenolics (trichlorophenol, pentachlorophenol)
- High Molecular Weight Aliphatic and Branch Chain Hydrocarbons (gasoline, kerosene)
- Chlorinated Aliphatic Hydrocarbons (carbon tetrachloride, perchloroethylene)
- High Molecular Weight Aliphatic Acids and Aromatic Acids (tar acids, 2,4-dichlorobenzoic acid, sulfonated lignins, benzoic acid)
- High Molecular Weight Aliphatic Amines and Aromatic Amines (aniline, toluene diamine)
- High Molecular Weight Ketones, Esters, Ethers and Alcohols (hydroquinone, polyethylene glycol)
- Surfactants (alkyl benzene sulfonates, linear alcohol sulfates)
- Soluble Organic Dyes (methylene blue, Indigo carmine, Benzopurpurin 4B Phthalocyanines)
INVESTMENT

Calgon supports its many pollution control service clients with the most advanced carbon reactivation facilities available.

Most economical technology. The economics of adsorption are related directly to the ability of granular carbon to be reactivated for re-use, thus lowering the costs.

Reactivation gives granular activated carbon an important economic advantage over the other methods of industrial wastewater treatment. This cost-saving feature of carbon has resulted in a growing trend to use it for municipal as well as industrial wastewater treatment.

After the carbon has adsorbed impurities, the granules can be restored to their original adsorptive capacity through thermal "reactivation". This process destroys adsorbed organic contaminants in the pores of the carbon granules. It is economically advantageous to use carbon adsorption at the point source to remove organic pollutants before they are diluted in the main plant wastewater stream. Considerable reduction can be effected in size of treatment facilities and the energy required to operate them when point source treatment is employed.

Another potential for economy is often possible through the recovery of water, heat energy or product. Savings of these types have been demonstrated many times in full-scale applications.

Calgon Corporation, a major producer of granular activated carbon, has developed the basic technology associated with the economical reactivation of granular carbon. Carbon reactivation facilities are often included as part of the basic wastewater treatment system in large-scale pollution control systems. However, the cost of on-site reactivation facilities for wastewater treatment systems using relatively small amounts of carbon is often prohibitive. As a result, the benefits of carbon treatment systems for smaller plants were tempered by the relatively high cost of reactivation.

Calgon overcame this problem through the introduction of its Calgon Carbon Service for water pollution control.

Most readily available technology. Calgon recognizes the need to provide practical solutions to pollution problems in the shortest possible time. If the testing program demonstrates that carbon will effectively remove organic contaminants from your plant's effluent, Calgon will take immediate action to have the complete treatment system installed in approximately 90 days from signing of a contract.

This quick installation is made possible through the use of modular systems which are inventoried for rapid shipment and installation. The client simply provides the necessary utility connections and a concrete pad for the equipment.

Calgon supervises the interconnection of treatment modules, puts the system on line, and can even operate it for you. The service fee includes installation and maintenance operations required for uninterrupted operation. It can also include analytical services required for the proper monitoring of results. Calgon supplies carbon for the system in trucks which are specially designed for simplified delivery and transfer of fresh carbon, and for the efficient removal of used carbon. The Calgon truck removes all exhausted carbon and transports it to a Calgon-operated facility for reactivation.

The system you are provided is essentially a closed system which does not create secondary air pollution control problems, sludge problems, or similar environmental problems.
...IN THREE EASY STEPS

COMPREHENSIVE PROBLEM DIAGNOSIS AND WASTEWATER EVALUATION.

1

With the assistance of a Calgon representative, you will be asked to provide information on the characteristics of the waste stream(s) to be treated. This information includes flow rate, concentration of suspended and dissolved solids and oil, a general description of the sources of waste, general identification of the types of organic material present in the stream, and a review of effluent requirements.

Next, you will be requested to supply a representative five-gallon sample of your plant's wastewater for analyses by Calgon. A suitable container and detailed directions will be provided by Calgon for this purpose. Calgon will then conduct feasibility tests in its laboratories to determine the amenability of your wastes to granular activated carbon treatment.

On the basis of these studies, the Calgon representative will advise you whether granular activated carbon adsorption is a cost/effective treatment method for you.

2

If the Calgon Carbon Service is a potentially cost/effective solution, Calgon will submit a detailed proposal covering the equipment and services it will provide. The system recommended for your plant will be based on the use of standardized treatment modules — a major cost-saving factor.

Included in the proposal will be a statement of services and equipment covered by the proposed service fee. It will detail areas of responsibility and specify the levels of wastewater treatment which are to be provided.

Acceptance of the proposal is contingent upon successful validation studies which Calgon will perform to confirm cost effectiveness of the Calgon Carbon Service.
DELIVERY, START-UP, OPERATION AND MONITORING.

A Calgon wastewater treatment system can be in operation at your plant more quickly than almost any other alternative system.

The only installation steps required at your plant are the preparation of the site, providing the necessary utilities for operation, and providing the piping system needed to bring wastewater to the treatment system and to discharge after treatment. Calgon will supervise the installation of the system, and can even operate it for you if desired.

Calgon will monitor plant wastewater before and after treatment on an agreed upon basis. Samples will be analyzed in the Calgon laboratories. You will be provided with reports of the test results.

Calgon assumes responsibility for maintenance of its equipment. Spent carbon will be removed from the plant site by Calgon personnel at regular intervals and reactivated. A full supply of carbon — more than enough to meet plant operating requirements — will be maintained in the system by Calgon at all times.

Start now! A letter or telephone call to Calgon will bring a prompt response from a fully-qualified carbon specialist. For fast and detailed information, write: Calgon Activated Carbon Division, Calgon Corporation, P.O. Box 1346, Pittsburgh, Pa. 15230; or call your nearest Calgon technical representative:

- Bridgewater, NJ  (201) 526-4646
- Pittsburgh, PA  (412) 777-8000
- Chicago, IL  (312) 325-9090
- San Mateo, CA  (415) 572-9111
- Houston, TX  (713) 682-1301
- St. Louis, MO  (314) 569-1599

A typical Calgon Carbon Service system can include the following modular units built with materials of construction consistent with corrosion considerations for each wastewater being treated:

1. Granular carbon adsorber modules
2. Filter modules
3. Carbon handling system
4. Pumps, controls and instruments
5. Prefabricated building

Granular Carbon Adsorber Modules
Systems ranging from single-state to multistage/multitrain will be provided according to your needs.

Filter Modules
Filters can be provided when suspended solids content is excessive. The type of filtration employed is dependent on type and quantity of suspended solids, and the availability of solids disposal facilities at the plant location.

Carbon Transfer Modules
Periodic carbon replacement is facilitated by means of an automated carbon handling system.

Pumps and Controls
Suitable pumps and flow indicator and controller can be provided.

Enclosure
Where required, Calgon can provide a suitable structure for enclosure of the carbon system with lighting, heating and ventilation included.
Carbon's effectiveness in the treatment of industrial wastewaters is directly related to its capacity for adsorption of organic molecules. Adsorption is a physical, rather than chemical or biological process. The phenomenon of adsorption is created by the presence of unbalanced physical forces (Van der Waals forces) in the surface of the carbon granule. Such forces have an affinity for organic molecules — including those frequently encountered in wastewater.

Although the unbalanced forces create some degree of attraction or "pull" at the exterior surface of the carbon granule, they are particularly strong within the individual pores of the granule. Here, the concentration of unbalanced forces around the pore walls creates an effective and powerful trap for the containment of organic wastes. Since each carbon granule has an extensive internal network of pores, carbon has a very high surface area suitable for the attraction and removal of impurities from water.

For further information write: Calgon Activated Carbon Division, Calgon Corporation, P.O. Box 1346, Pittsburgh, Pa. 15230; or call your nearest Calgon representative:

- Bridgewater, NJ  (201) 526-4646
  Pittsburgh, PA  (412) 777-8000
- Chicago, IL  (312) 325-9090
  San Mateo, CA  (415) 572-9111
- Houston, TX  (713) 682-1301
  St. Louis, MO  (314) 569-1599

CALGON CORPORATION
SUBSIDIARY OF MERCK & CO., INC.
Appendix J-7

SHIRCO INCINERATION REGENERATION SYSTEM COSTS

Operating Costs

Utilities, Labor, Maintenance........ $0.21/lb
Extra Maintenance due to hi HCL....... $0.10/lb
Carbon, 10% loss at $1.00/lb.......... $1.10/lb

TOTAL.................................. $4.77/lb

Investment

Shirco Unit (100 lb/hr)............... $400,000
Utilities, Site Preparation........... $200,000

TOTAL.................................. $600,000
Appendix J-8

COST COMPARISON OF OPTION 3 (GRANULAR CARBON - PBA REGENERATION) VS
OPTION 2 (GRANULAR CARBON - CONTRACT REGENERATION)

<table>
<thead>
<tr>
<th>Flow Rate GPD</th>
<th>Granular Carbon Loading, g/g</th>
<th>Option 2 Cost $/yr</th>
<th>Option 3 Cost $/yr</th>
<th>Option 3 Savings $/yr</th>
<th>Payout, Years</th>
</tr>
</thead>
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<tr>
<td>250,000</td>
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</table>

Appendix J-6

Carbon Regeneration by Contractor Service (Calgon Corp)
Appendix K

Sludge Removal Systems
Prior to Carbon Column Treatment
Appendix K-1

Existing PBA Waste Settling Lagoons and Treatment Systems
Appendix K-2

Parkson Sludge Dewatering Press
Appendix K-3

Lamella Settler
The "LGST" is a high-rate gravity settler combined with a circular picket-fence sludge thickener/scaper in one tank. The high-rate settler section utilizes the same counter-current flow principle and inclined-plate design that is currently used in Parkson's packaged Lamella Gravity Settlers. Both 45° and 55° plate angles are available.

LGST gives you these benefits:

- Space requirements are reduced by 90% or more, resulting in lower installed costs.
- Underflow sludge concentrations are up to five times higher.
- Wind and thermal currents are virtually eliminated, thus overflow quality remains excellent year-round, and there is no sludge blanket to maintain.
- Eliminates the need for a second stage thickener and related flocculants (if required).
- Significant cost reductions for dewatering equipment and conditioning chemicals when hydroxides are being settled out. Savings can be up to 50% or higher.

The LGST is designed to:

- Handle flow rates and/or solids loadings beyond that which the package Lamella Gravity Settler units can handle economically; and/or
- Produce unprecedented sludge concentrations by gravity when hydroxide-type solids are being settled out; and
- Provide sludge storage for flexibility in sludge dewatering equipment operations.
The LGST Design Consists Of:

**LGST Plate Pack**

The influent stream enters the plate packs through a series of openings on both sides of the packs. The feed flows onto the plates from each side and then upward exiting through a series of flow distribution orifices into the effluent flumes. These orifices are sized to take a specific pressure drop on the clarified effluent to ensure uniform flow distribution between each pair of plates. The solids settle out onto the plate surfaces and slide downward into the thickening compartment.

**Picket Fence-Type Sludge Scraper**

It has been known for some time that gentle stirring during the thickening/compression stage is an effective aid to concentrating sludges. A specially designed version of a conventional picket-fence thickening/scaper is utilized to produce this stirring in the "LGST".
Appendix K-4

Solids/Sludge Concentration With Hydrocyclones
Facts about liquid cyclones. Where to use them. Where not to use them. And how to specify the right cyclone for the job.

With special emphasis on the Dorrclone® family of cyclones from Dorr-Oliver for a broad range of applications.
The liquid cyclone. What it is.
A processing device that uses pressure energy to create rotational motion in a body of liquid... thus generating a centrifugal force that separates one material from another. Solids from liquid. Coarse particles from fine.

The liquid cyclone as a process tool.
The cyclone performs a wide range of separation functions. You should consider its use in your process wherever the separation of materials is required.

The DorrClone can be used instead of gravity equipment.
In many processes it performs comparably to gravity devices. In other processes it offers a more economical or more practical way of doing the job.

The DorrClone can be used in combination with other equipment.
It should be used ahead of high speed equipment to protect it from abrasive wear. It will also enable the use of smaller machines by reducing the load.

The DorrClone should be considered as a primary—or supplementary—process stage in all of the following:
- Classification
- Degritting
- Closed-circuit grinding
- Desliming
- Concentration
- Recovery of solids
- Countercurrent washing
- Clarification

The science lies in knowing where to use the DorrClone cyclone. Where not to use it. And how to specify the right one for the job.

That is what this book is all about.
How the DorrClone® works. And makes a separation.

Basically the DorrClone separator is a cylindrical/conical shell with a tangential inlet for feed... and outlets for both an underflow and an overflow.

The flow pattern—the principle upon which any liquid cyclone operates—is shown in the diagram at the left.

The feed—which is predominantly liquid—is injected into the unit tangentially. With sufficient velocity to create a vortex action.

The vortex action forces the liquid into a spiral. And—as the rapidly rotating liquid spins about the axis of the cone—it is forced to spiral inward... and out through the centrally located overflow outlet.

Larger heavier particles of solid matter are thrown outward against the wall of the cone by the centrifugal force within the vortex. These particles spiral around the wall of the cyclone and exit through the apex of the cone as underflow.

Smaller particles remain in the liquid as it spirals inward. And are discharged with the overflow.

Controlling the separation.

The cyclone is essentially a simple instrument. The science of using it effectively is to control certain design variables to make the unit do the job you want it to do.

For example. Larger diameter cyclones produce coarser separations. And longer cyclones produce finer separations.

Cyclones, in short, can be designed to produce separations of a predetermined fineness, coarseness or sharpness.

The separation. Some definitions.

Coarse and fine refer respectively to the "large" particles reporting to the underflow and the "small" particles reporting to the overflow.

The separation refers to the solid particles reporting to the underflow.

The size of the separation is a measure of the diameter of the 95% particle (d₉₅), the particle which has reported to the underflow in 95% of its total quantity. (An alternate method is based upon the d₉₀ particle, half of which reports to overflow and the other half to underflow. The d₉₀ particle is usually about 0.55 of the size of the d₉₅ particle.)

Most performance charts further define the separation in terms of the following: (1) Particles that are roughly spherical in shape. (2) 2.7 specific gravity particles suspended in water at 20°C.

Sharpness of separation refers to the fact that no cyclone separation is perfect. There will always be some fines in the underflow, some coarse in the overflow. The sharper the separation, the fewer the fines in the coarse; the fewer the coarse with the fines. Sharpness of separation generally depends upon the feed solids size distribution and how far apart the designated fractions are in size or weight, or on solids underflow concentration and d/D ratio.

Product is whatever you wish to retain or recover. It can consist of either the underflow or the overflow. It can be contained in underflow or overflow and recovered in subsequent separation processes.
Basic DorrClone construction. And how "design variables" regulate the separation.

The DorrClone cyclone performs as a classifier because materials that are different in size, shape or density report to different ends of a cyclone. Regulation of certain "design variables" controls this separation effect.

For example, the most basic of the design variables is the diameter of the cyclone itself.

A cyclone of a specific diameter cannot produce an underflow containing a majority of solids smaller than a specific size. Smaller cyclones must be used to capture smaller solids.

Division of separation, therefore, can be predetermined. A cyclone of properly specified diameter will capture solids larger than a certain size in the underflow. And discharge solids smaller than that size in the overflow.

Other design variables regulate the separation in similar manner. And the interrelation of all of the design variables determines the precise quality of the separation.

Any cyclone separation is a function of the design variables shown in diagram on page 7. These design variables include:

1. **Cyclone diameter.** The larger the diameter, the coarser the separation. The smaller the diameter, the finer the separation.
2. **Feed inlet diameter.** Within certain limits, the larger the inlet, the greater the cyclone capacity, the coarser the separation. The smaller the inlet, the lesser the capacity, the finer the separation.
DorrClone concentrates salt crystals for feed to Mercon centrifuge at a chemicals plant.

The DorrClone
Where to use it

General advantages.
All types of DorrClone.

High capacity.
The basic efficiency of the cyclone means that individual units can handle exceptionally large flow volumes. Units of 18-inch diameter, for example, customarily produce throughputs of up to 1200 gpm, depending upon inlet pressure. The potential of grouping clusters of DorrClone cyclones in manifolds means that their effective capacities are virtually unlimited.

Lower processing costs.
DorrClone separators can save in two ways. They can replace a more costly gravity-type classifier. Or they can reduce wear and tear on existing process equipment through pre-treatment of slurry.

Simplicity of design.
Low maintenance.
Lack of moving parts mean minimum maintenance. Liners—where used—designed for economical replacement. And all DorrClone models are designed for simple, efficient servicing.

Compact, economical installations.
Compact design requires less costly floor space. DorrClone stations can be installed in "tight" locations where conventional equipment would not fit.

Versatility in application.
There are many types of DorrClone available. And each is designed for a wide range of uses.
in your process. Why use it. Where not to use it.

**Predictable performance.**
The cyclone is essentially a simple instrument. It performs a precision function, however, and must be scientifically engineered and manufactured to exacting standards. Dorr-Oliver has been producing quality cyclones since 1948.

**Assistance from Dorr-Oliver.**
Dorr-Oliver does more than offer a full line of cyclone separators. We provide technical data. And whatever technical assistance you may require in effectively integrating the DorrClone into your flow sheet.

**Low equipment investment.**
The cost of installing a DorrClone station is relatively small. Operating costs are very low.

**Spec. job advantages for specific jobs.**
Each type of DorrClone is designed to fulfill certain demands. Each offers individual advantages in addition to the general advantages listed above. See the next page for a breakdown of DorrClone types.

**When not to use a DorrClone.**
- When the coarse solids to be removed are less than 5 microns in size. Although there are exceptions, this is normally impractical.
- When there is insufficient specific gravity differential between the two fractions you wish to separate.
- When two solids fractions have the same settling characteristics.
- When the liquid in the slurry is of high viscosity.
- When a dry underflow is essential. (The underflow from any DorrClone separator requires liquid media in order to discharge.)
- When a properly executed Problem Analysis Worksheet indicates lack of feasibility.

**Industries in which DorrClone has proven itself as a classification tool.**
- Minerals processing.
- Chemical processing.
- Food processing.
- Pharmaceutical processing.
- Petroleum and petrochemicals.
- Pulp and paper.
- Waste and water treatment systems.
Particle Removal

Numbers are particle diameter size in microns, 95% of which are removed in the underflow at the indicated capacity and pressure drop. Separations are based on 2.7 Sp.G. solids at feed concentration below 25% by weight.
A guide to DorrClone® specification.

The charts shown below will allow you to make some preliminary judgments. As to whether or not a DorrClone cyclone will add to the efficiency of your process. As to what type and size of DorrClone will do the job most effectively.

To use the charts it is required that you know the particle size of your separation in microns. For final and accurate specification refer to the Problem Analysis Worksheet on page 19.

![Diagram of operating pressure drop in PSIG versus difference between inlet and overflow pressure with labels for 10 MM and P-25.]
Technical Data Sheets.
Individual DorrClone Types following pages.
Typical Flowsheets on this page.
Appendix K-5

Pilot Hydrocyclones for Test and Evaluation of
Sludge/Solid Concentration
Mozley Hydrocyclones

Mozley Hydrocyclones are manufactured in wear-resistant polyurethane. They are supplied with a range of easily interchangeable vortex-finder and spigot caps having different outlet diameters enabling cut points from 2 to 15 microns to be achieved.

**Mode of Operation**

Feed slurry enters the hydrocyclone tangentially under pressure. As a result of the high centrifugal forces, particles coarser than the 'cut point' migrate into a primary vortex adjacent to the wall and move downwards to discharge with a small volume of water via the spigot. Particles finer than the cut point migrate into a secondary upward-moving vortex, along the axis of the hydrocyclone, and discharge with the majority of the water via the vortex finder.

**Particulate Finer than Cut Point**

Particles finer than 100 microns in water.

**Spigot**

Easily removable spigot cap. UNDERFLOW: Particles coarser than cut point plus water.

**Overflow**

Water plus particles finer than cut point.

Mozley 1" and 2" Hydrocyclones are not normally recommended for treating slurries containing significant quantities of particles coarser than 100 microns.
Performance characteristics

Applications

The performance of a given hydrocyclone depends on the operating pressure and the diameters of the outlets. In general, a higher operating pressure will give a higher throughput, and a sharper cut at a finer cut point. The graphs below show throughput against operating pressure for single Mozley Hydrocyclones, and typical efficiency curves at normal operating pressures - it should be noted that the $d_{50}$ for each hydrocyclone size can be varied between half and twice the value shown, by selecting appropriate pressures and outlet diameters. The table shows the volume splits to underflow at normal operating pressures.

Assemblies housing 2 to 40 hydrocyclones are available to handle large throughputs. To calculate the number of hydrocyclones required to treat a given flow, take the throughput of a single hydrocyclone at the design pressure and divide the total flow by this throughput.

Mozley hydrocyclones have been used successfully for making fine separations in a wide range of industrial applications.

MINING
Preparation of tin flotation feed
Recovery of fine potash
De-watering tailings streams
Beneficiation of china clay (kaolin)
Recovery of fine marble and gypsum
Classification of fine table feeds
Recovery of fullers earth from organic solvent
De-sanding tantalite tailing prior to flotation
Preparation of scheelite ore

ENGINEERING
Removal of oil from machine coolants
Classification of metal powders
Removal of fine swarf from cutting fluids
Recovery of white metal particles from lubricant

MANUFACTURING
Recovery of grinding media in glass manufacture
Classification of pigment suspension
Cleaning up recycled water in high pressure jetting equipment
Recovery of activated carbon from oil solvent mixture
De-watering tallow after washing operation

POWER
Removal of fine solids from cooling tower discharge
De-watering coal flotation concentrate
Recovery of fine pulverised coal

EFFLUENT CONTROL
Removing starch from potato processing plant effluent
Cleaning chalk clay filtrate in cement manufacture
Recovery of copper particles from circuit board manufacturing effluent

OIL
Density control of drilling mud
Removal of cuttings from drilling mud
Cleaning completion fluids

CHEMICAL
Thickening tricalcium phosphate acetic acid slurry
De-gritting gas scrubber effluent ahead of centrifuge
Thickening centrifuge feed
Separating iron from iron oxide
Thickening dicalcium phosphate dihydrate slurry
Washing plastic granules in manufacturing process
Recovery of ammonium chloride crystals from mother liquor

FOOD
De-gritting parsley
Recovery of fine starch

Richard Mozley Limited
Woodlane - Falmouth
Cornwall - TR11 4RF - England
Telephone: (0326) 313260/316021

DISTRIBUTED BY:
CARPDCO, INC.
4120 FLAMINGO STREET
JACKSONVILLE, FLORIDA 32206 U.S.A.
TELEPHONE: (904) 333-3681
TELEX: 5-6357 CARPDCO JAX
GENERAL:

The purpose for the hydrocyclone test rig is to provide the capability to evaluate the operating parameters for hydrocyclones for separating solids from various wastewaters. The hydrocyclone test rig supplied shall be suitable for the intended purpose. The test rig supplied shall include, but not limited to, all the equipment and supplies indicated on sketch, Figure 1, and indicated in these specifications. The test rig supplied shall be completely assembled when shipped.

The test rig shall include, but not be limited to, two hydrocyclones with accessories, slurry sump, pump, pump drive motor, motor control box, support vehicle and frame, pressure gauge, piping and valves. The slurry sump shall be located so the underflow and overflow from hydrocyclones will be collected in the sump. The hydrocyclones shall be constructed with tangential feed inlet.

HYDROCYCLONES:

The test rig supplied shall include two hydrocyclones. One of the hydrocyclones shall be 2" in diameter and one of the hydrocyclones shall be 10mm in diameter. The hydrocyclones shall be constructed of transparent material so that vortex can be observed. The 2" diameter hydrocyclone shall be supplied with interchangeable vortex finders of 5/16", 7/16", 9/16" diameter and interchangeable underflow spigots of 1/8", 3/16", 1/4", 5/16" diameter.

The 10 mm diameter hydrocyclone shall be supplied with interchangeable vortex finders of 2mm, 2.3mm, 2.6mm diameter, and a fixed underflow spigot of 1/2mm. The hydrocyclones shall be constructed so that the vortex finders change can be easily changed, and the two different diameter hydrocyclones can be easily interchanged and attached to the remainder of the test rig.

PUMP:

The test rig shall be supplied with a progressive cavity pump capable of supplying 20 GPM water at 65 psig. The pump shall be powered with a 2 HP TEFC 60 Hertz 115 VAC single phase electric motor. The motor shall be coupled to the pump with pulleys and belt(s) with provisions for changing pulleys to change pump speed or be coupled to pump with direct drive with provisions for changing pump speed. A NEMA4 control box shall be supplied with the motor and shall include stop-start switch with circuit breaker. A 13 prong 115V AC electrical cord shall be used to supply power to the motor control box.

SLURRY SUMP:

The test rig shall be supplied with a funnel shaped slurry sump connected to pump inlet. The slurry sump shall be approximately 21" in diameter and house a capacity of approximately 5 gallons.
SUBJECT: Specifications for Hydrocyclone Test Rig

PIPE, VALVES, HOSE, GAUGE:

The test rig shall be supplied with the pipe and valves indicated in Figure 1. The test rig shall be supplied with a pressure gauge with range from 0-100 psig. A flexible hose shall be supplied to return the hydrocyclone overflow to the slurry sump.

SUPPORT VEHICLE AND FRAME:

The test rig shall be supplied with a support vehicle, as indicated in Figure 1, to provide portability to test rig. The support vehicle shall include the support base with wheels, retractable legs and handle. A frame shall be attached to the support base for supporting the hydrocyclones and other equipment as necessary.
Appendix K-6

Critical Evaluation of the Simple Ways To Determine Hydrocyclone Cut Size
CRITICAL EVALUATION OF THE SIMPLE WAYS OF DETERMINING THE CUT SIZE

L. Svarovský
University of Bradford, U.K.

Summary

Efficiency of separation of hydrocyclones is best described by the full grade efficiency curve. For simplicity, however, the "cut-size" has been used as a single number characterizing a hydrocyclone operated under given conditions. Basically, three types of different cut sizes are currently in use in industry: the equiprobable size, the analytical cut size and the curve intersection cut size. In most applications, the three cut sizes obtained are different in value and to a varying degree dependent on the size distribution of the feed.

The objective of the present study is to show the effect of feed size distribution on the cut sizes and to provide some guidance to the practising engineer as to what correction he may apply to minimize this effect.

The paper starts with the definitions and summary of the simple ways of determining the three types of cut size. It goes on to compare the three in both their simplicity of use and how they are affected by the size distribution of the feed material. While the equiprobable size is the best of the three, the analytical cut size is simpler in evaluation and use, and is always closer to the former than the intersection cut size.

The paper then concentrates on the analytical cut size in order to quantify its deviation from the equiprobable size and goes on to derive a simple correlation which can be used to convert it into the equiprobable size (which is then independent of the feed distribution).

This correlation is obtained for the most frequent shape and slope of the grade efficiency curve and the error margins are shown for other slopes. It therefore provides a method of determining the equiprobable size from much simpler tests than normally required. It also allows a more reliable prediction of total efficiency from the known cut size. The application of this method is shown in practical examples on classification of powders or solid-liquid separation with hydrocyclones using published test information.
**NOMENCLATURE**

- $A_1, A_2, A_3, A_4$: constants
- $B_1, B_2$: constants
- $C_x, C_u$: feed concentration
- $E_T$: underflow concentration
- $E_r$: total efficiency
- $R_T$: reduced total efficiency
- $F$: cumulative size distribution in feed
- $F_x$: cumulative size distribution in underflow
- $F_r$: cumulative size distribution in overflow
- $G$: grade efficiency
- $G_r$: reduced grade efficiency
- $R_x$: underflow-to-throughput ratio
- $Q$: feed flowrate
- $Q_x$: underflow rate
- $X$: particle size, variable
- $X_a$: analytical cut size
- $X_m$: median size
- $X_1$: intersection cut size
- $X_{50}$: equiprobable size (cut size)
- $x_{50}$: reduced cut size
- $y$: variable
- $s$: variable
- $\sigma_g$: geometric standard deviation of solids distribution
- $\sigma_{gs}$: geometric standard deviation of grade efficiency curve
1. INTRODUCTION

The efficiency of separation of hydrocyclones is highly size-dependent and is best expressed as the grade efficiency curve \( G(x) \). This curve, often also called the Trump curve, gives the probability of separation of different particle sizes into the underflow and is typically S-shaped - see Figure 1.

Expressing grade efficiency as a graph, a table of values or as an analytical function is often too clumsy for correlation against operating variables or even for a simple equipment comparison. Such cases call for a single number, independent of the feed size distribution, as a measure of efficiency. This is available in the form of the cut size which is the subject of the study presented here.

The concept of cut size is based on a simple analogy with screening: the cut size is the aperture size of a hypothetical and ideal screen which would achieve the same separation as the hydrocyclone. This is a simple enough analogy but the problem is that the separation performance of a hydrocyclone is very different from that of an ideal screen.

An ideal screen (a screen with precisely uniform size of the apertures and infinite time of screening) has a grade efficiency in the form of a step function (Figure 1) while a hydrocyclone gives much less sharp separation due to uneven conditions to which different particles are subjected in a hydrocyclone. This is reflected into the grade efficiency curve which becomes a curve sloping upwards at an angle of much less than 90°. This curve should go through the origin because, due to the predominant inertial separation in a hydrocyclone, \( G(x) \) should be zero for \( x = 0 \) but this is not true as a finite amount of liquid has to be discharged with the solids through the underflow and this liquid contains solids of all sizes.

The presence of flow splitting or "dead flux", particularly in applications with appreciable and dilute underflow, modifies the shape of the grade efficiency curve and makes it appear as if the separator performs better than is due to the net separation process. The effect can be seen in Figure 1, where the grade efficiency curve of a hydrocyclone has an intercept. The apparently finite efficiency for particles approaching zero in size is in fact equal to the underflow-to-throughput ratio \( R_f \) under which the hydrocyclone was tested, because the very fine particles simply follow the flow and are split in the same ratio as the liquid.

In order to remove this "guaranteed" efficiency from equipment comparisons, the grade efficiency definition is "reduced" in the following way:

\[
G'(x) = \frac{G(x) - R_f}{1 - R_f}
\]

The curve based on this definition does go through the origin indicated by the dashed line in Figure 1; the size corresponding to 50% is the reduced cut size \( x_{50} \). The total efficiency \( E_T \) is reduced to \( E_T' \) in the same way as the grade efficiency \( G(x) \) in Equation (1).

2. DEFINITIONS OF CUT SIZE

The best definitions of cut size are derived from the grade efficiency curve and characterize the relative position of the curve along the x-axis.

The so-called equiprobable size \( x_{50} \) is perhaps the most logical and most widely used cut size; it is the size corresponding to 50% on the grade efficiency curve. It is therefore the particle size which has equal probability of reporting to the underflow or to the overflow. In other words, all particle sizes in the feed above \( x_{50} \) will be mostly separated and those below will be mostly unseparated.

The most obvious way of determining \( x_{50} \) is from its definition, which requires evaluation of the grade efficiency curve. Alternatively it can also be determined from a simple plot of a composite function of the test data where it corresponds to...
the maximum (1). In either case the measured data needed for such an exercise comprises the total mass recovery and the size distributions of the solids in two out of the three material streams involved or, alternatively, the size distributions in all three streams.

In industrial practice the above listed data may not always be easily obtainable and the tendency has been to use other definitions of cut size, the evaluation of which require less effort and fewer measurements. The two other definitions listed below are most common in practice.

The so-called analytical cut size $x_a$ is the size which would ideally split the feed solids according to particle size in proportions given by the measured total efficiency $E_T$. It is therefore the size corresponding to the percentage equal to $E_T$ on the cumulative particle size distribution oversize $F(x)$ of the feed solids (Figure 2). Thus:

$$F(x_a) = E_T$$

The analytical cut size is therefore the aperture size of a hypothetical and ideal screen which would give the same mass recovery of solids $E_T$ as the hydrocyclone. It also follows from this definition that, for practical unsharp classification, at the analytical cut size the total amount of misplaced material in the two products is equal (i.e. that the total mass of solids finer than $x_a$ in the coarse product is equal to the total mass of solids coarser than $x_a$ in the fine product). This is of course very rarely true about the equiprobable size $x_{50}$ and the two generally do not coincide.

Another cut size is defined (Ref. 2) as the size $x_i$ at which the cumulative percentage undersize of the coarse product (separated solids in the underflow) is equal to the cumulative percentage oversize of the fine product (unseparated solids), or vice versa (Figure 3). The point corresponding to $x_i$ is clearly determined from the intersect of the two curves representing the two products, without any need for the knowledge of the total efficiency $E_T$. The value of the cut size derived in this way is even more sensitive to the changes in the feed size distribution than the analytical cut size, as can be seen from the discussion in the following section.

Mathematically, the intersection cut size $x_i$ is defined as:

$$F_C(x_i) = 1 - F_F(x_i)$$

where $F_C$ and $F_F$ are the percentages undersize of the coarse and fine products respectively.

3. RELATIONSHIPS BETWEEN THE THREE CUT SIZES

As the equiprobable cut size $x_{50}$ is derived from the grade efficiency curve, it is in most applications independent of the size distribution of the feed solids and is therefore most suitable as a simple measure of hydrocyclone efficiency under given operating conditions.

The values of the other two commonly used cut sizes, the analytical cut size $x_a$ and the "curve intersection" cut size $x_i$, are always different from $x_{50}$, unless $x_{50}$
is equal to the median of the feed size distribution \( x \) when all three coincide; the difference between \( x_g \) and \( x \) increases as the median of the feed moves towards either end of the grade efficiency curve. This is best shown in the plot of \( G(x) \) and \( F(x) \) in Figure 4 from which it can be seen that both \( x \) and \( x_g \) are always on the same side of \( x_{50} \) as the median \( x_g \) of the feed, with \( x_a \) being always closest to \( x_{50} \), hence:

- If \( x_g < x_{50} \) (i.e. fine feed, curve A in Figure 4)
  \[
  x_g < x_1 < x_a < x_{50}
  \]

- If \( x_g = x_{50} \) (curve B in Figure 4)
  \[
  x_g = x_1 = x_a = x_{50}
  \]

- And if \( x_g > x_{50} \) (i.e. coarse feed, curve C in Figure 4)
  \[
  x_g > x_1 > x_a > x_{50}
  \]

The above relationships can be proved mathematically but just a diagrammatic presentation in Figure 4 is sufficient here. For example, as the total efficiency for the curve A is represented by the area under the curve (see Reference 2) then it satisfies the condition in Equation (2) the analytical cut size \( x_a \) is that point on curve A which corresponds to a rectangle of equal area determining \( F(x_a) \) on the x-axis. The intersection cut size \( x_1 \) can be shown to correspond to the following geometric condition: if the total area under the curve is \( A_1 = F(x_a) \) and the partial area to the left of \( F(x_1) \) is \( A_1 \), and if \( B_1 \) is the total area above the curve \( (1-F(x_a)) \) with \( B_2 \) being the fraction to the right of \( F(x_1) \) then the position of \( F(x_1) \) is such that it satisfies the following condition:

\[
\frac{A_1}{A_1} = \frac{B_1}{B_2}
\]

4. CONVERSION OF \( x_a \) INTO \( x_{50} \) AND VICE VERSA

Theoretical conversion of the easily obtainable analytical cut size \( x_a \) into the equiprobable size \( x_{50} \) is possible if both the feed size distribution and the grade efficiency curve can be approximated by an analytical function.

One such conversion is given below for the case of both of the functions being log-normal.

Many practical particle size distributions approximate the log-normal distribution in the form:

\[
\frac{dp}{dx} = \frac{1}{\ln g \sqrt{2\pi}} \exp \left( -\frac{(\ln x - \ln x_g)^2}{2 \ln^2 g} \right)
\]

where \( x \) is the median (=geometric mean) and \( \sigma_g \) is the geometric standard deviation; both of those can be easily determined from a cumulative plot in a log-probability paper (Ref. 2).

Integration of Equation (6) yields the cumulative form of log-normal distributions (fraction oversize):

\[
F(x) = \frac{1}{2} - \frac{1}{2} \text{erf} \frac{\ln x - \ln x_g}{\sqrt{2 \ln^2 g}}
\]
The erf function is defined as:

\[
\text{erf}(z) = \frac{2}{\sqrt{\pi}} \int_0^z e^{-t^2} \, dt
\]  

(8)

and can be solved by using standard tables or using an analytical approximation such as the one given in the Appendix.

Interestingly enough, some practical grade efficiency curves can also be fitted by the cumulative log-normal distribution so that similarly to Equation (7) (but this time fraction "undersize"):

\[
G(x) = \frac{1}{2} + \frac{1}{2} \text{erf} \left( \frac{\ln x - \ln x_{50}}{\sqrt{2} \ln g} \right)
\]  

(9)

where \(x_{50}\) is the equiprobable size and \(g\) is the geometric standard deviation of the curve.

The total efficiency \(E_T\) is determined from \(G(x)\) and \(F(x)\) by integration (Ref. 2):

\[
E_T = \int_0^1 G(x) \, dx
\]  

(10)

and this using Equations (7) and (9) yields (Ref. 3):

\[
E_T = \frac{1}{2} + \frac{1}{2} \text{erf} \left( \frac{\ln x - \ln x_{50}}{\sqrt{2} \ln^2 g + \ln^2 g} \right)
\]  

(11)

where \(x\) and \(g\) refer to the feed size distribution and \(x_{50}\) and \(g\) refer to the grade efficiency function.

The final conversion formula is obtained from the definition of the analytical cut size in Equation (2) by substitution of \(F(x)\) from Equation (7) and \(E_T\) from Equation (11) as follows:

\[
\frac{x_{50}}{x} = \sqrt{1 + \left( \frac{\ln g_{50}}{\ln g} \right)^2}
\]  

(12)

The above equation can be used to calculate the true cut size \(x_{50}\) from the known size distribution of the feed defined by \(x\) and \(g\) and from the simply measured analytical cut size \(x\) providing that an estimate of the geometric standard deviation \(g\) of the separator's grade efficiency curve is available. Such information can be found in the literature. Gibson (Ref. 4), for example, has recently given values of \(g\) for a disc centrifuge and a hydrocyclone under different operating conditions.

Better still, Equation (11) can be used directly to calculate \(x_{50}\) from \(E_T\), but this involves the inverse of the error function. Appendix should be consulted as to how this can be solved.

For the reverse problem of estimating the total mass recovery \(E_T\) from \(G(x)\) and \(F(x)\) this is easily done using Equation (11); the corresponding analytical cut size can be calculated either from Equation (7) for \(F(x) = E_T\) or from \(x_{50}\) using Equation
With hydrocyclones, the log-normal function can only be fitted to the reduced grade efficiency curves (Ref. 4), that is, after elimination of the dead flux effect but this presents no problem because the conversion given above is then simply based on the reduced cut size $x_{50}$ instead of $x_{50}^*$.

Equation numbers (2), (4), (5), (9), (10), (11) and (12) are then applicable to the reduced grade efficiency $G'_v(x)$ and reduced cut size $x_{50}$, this is best shown in a numerical example in the following.

5. NUMERICAL EXAMPLE

Problem 1

Determine the reduced cut size $x_1$ of a 2" dia Moxy hydrocyclone fitted with a vortex finder of 7/16" dia and underflow spigot of 1/8" dia, if the following volumetric flowrates and solids concentrations were measured and if the feed solids-size distribution $F(x)$ followed a log-normal law (Equation (7)) with $x_g = 9$ µm and $\sigma_g = 2.0$:

- Feed flowrate $Q = 6.35 \times 10^{-4}$ m$^3$/s
- Feed solids concentration $C_f = 30$ kg m$^{-3}$
- Underflow rate $U = 3.5 \times 10^{-5}$ m$^3$/s
- Underflow concentration $C_u = 314.2$ kg m$^{-3}$

For the above cyclone Gibson (Ref. 4) found $\sigma_g = 1.75$.

Solution 1

By definition the total mass recovery of solids is:

$$E_T = \frac{C_u U}{Q C_f} = \frac{314.2 \times 3.5 \times 10^{-5}}{30 \times 6.35 \times 10^{-4}} = 0.5773$$

The reduced total efficiency is by Equation (1).

$$E_T^1 = \frac{E_T - R_f}{1 - R_f} = \frac{0.5773 - 0.0551}{1 - 0.0551} = 0.5526$$

as:

$$R_f = \frac{U}{Q} = \frac{3.5 \times 10^{-5}}{6.35 \times 10^{-4}} = 0.0551$$

Equation (7) for $F(x_a) = E_T^1$ gives $x_a = 8.21$ µm and Equation (12) yields $x_{50}^1 = 8.00$ µm.

Problem 2

What would be the estimated mass recovery $E_T$ and the analytical cut size $x$ with the same hydrocyclone under the same operating conditions if the same solids are treated but of a different size distribution characterised by $x_g = 10$ µm and $\sigma_g = 1.9$.

Solution 2

The reduced efficiency $E_T^1$ is directly from Equation (11) ($x_{50}^1 = 8$ µm, $\sigma_g = 1.75$).

$$E_T^1 = 0.6034$$ and if $R_f$ is the same (0.0551) then from Equation (1) it...

\[ A_{23} \]
The analytical cut size is from Equation (12):

\[ x_a = 8.45 \mu m \]

As predicted in Section 3, with increased \( x \) the analytical cut size moves further away from \( x_{50} = 8 \mu m \) (for \( x = 9 \mu m \) it was \( x_a = 8.21 \mu m \) while for \( x = 10 \mu m \), \( x_a = 8.45 \mu m \)).

6. Error Margins

The best way to get a simple idea about the errors in estimation of \( x_{50} \) from \( x \) as a result of errors in prediction of \( g'_s \) is from a plot of Equation (12) for different values of \( \sigma \). As can be seen from Figure 5, where a family of curves is plotted for equal increments of \( \ln (x_{50} / x) \) for a given dimensionless value of \( x / x_a \), the curves are practically equi-distant. This means that the error in \( x_{50} \) is approximately proportional to the error in \( \sigma \), with the coefficient of proportionality depending on \( x / x_a \). As could be expected the errors are minimum at the two extreme cases of the feed being much coarser (or finer) than \( x \) (i.e. \( x / x_a \to 0 \)), and of the feed \( x \) being nearly equal to \( x_a \) (and therefore also to \( x_{50} \)) so that \( x / x_a \to 1 \).

It is also clear from Figure 5 that however bad an estimate of \( \sigma \) it can never lead to the reversal of the inequalities of Equation (4). Uncertainty in \( g'_s \), if it can be estimated, is easily projected into uncertainty in \( x_{50} \) using Figure 5.

7. Conclusions

The two cut sizes, i.e. the analytical cut size \( x \) and the intersection cut size \( x_v \), not based on the grade efficiency curve vary with the fineness of the feed but are very much easier to obtain than the equiprobable size \( x_{50} \). It is shown here that \( x \) and \( x_v \), swing round \( x_a \) depending on whether the feed is coarser or finer than \( x_{50} \). This makes the use of \( x \) and \( x_v \) in place of \( x_{50} \) rather doubtful. However, if something is known about the expected slope of the grade efficiency curve at least the analytical cut size \( x \) can be converted to \( x_{50} \) (or be moved close to \( x_{50} \)). One such simple conversion is derived here which assumes both the solids size distribution and the expected grade efficiency curve as log-normal functions. Admittedly, this may not be in some cases valid but even if it is only very approximate it is invariably better to attempt a conversion than to blindly assume that \( x = x_{50} \). No conversion is needed either if the geometric mean of the feed is close to \( x_{50} \), i.e. when the reduced total mass recovery is around 50% or if the grade efficiency is very steep and \( \sigma \) therefore approaches 1.

There is no simple conversion of \( x \) into \( x_{50} \) along similar lines as for the analytical cut size \( x_a \).

APPENDIX

The error function defined in Equation (8) and used in Equation (7), (9) and (12) and whenever the cumulative form of the log-normal distribution is needed, can be evaluated manually from tabulated values or by time consuming computer approximations. Some relatively simple analytical approximations also exist which give excellent accuracy for engineering calculations. Hastings (Ref. 5), for example, published the following expression:

\[
\text{erf}(x) = 1 - (a_1 + a_2 x^2 + a_3 x^4) e^{-x^2}
\]  \hspace{1cm} (A1)
where \( t = 1/(1 + a x) \). The absolute error between the values from this expression and the tabulated values is less than \( 2 \times 10^{-3} \) with the following constants:

\[
\begin{align*}
 a_1 &= 0.3480242 \\
 a_2 &= -0.958798 \\
 a_3 &= 0.7478556 \\
 a_4 &= 0.4704700
\end{align*}
\]

The function in Equation (A1) is simple enough even for a programmable calculator. It does not however give a simple explicit form for the inverse of \( \text{erf} \) which might be needed to calculate \( x \) from Equation (7) or any other size corresponding to a known cumulative percentage \( F(x) \). For this purpose Karlsson and Bjerle (Ref. 6) have recently proposed another approximation so that if:

\[
\text{erf}(x) = y
\]

then:

either \( \text{erf}^{-1}(y) = \arcsin \arcsin \left( \frac{y}{\sqrt{2}} \right) \) (A2)

for \( 0 \leq y \leq 0.84 \)

or \( \text{erf}^{-1}(y) = (\arcsin y)^{1.5} \)

for \( 0.84 \leq y \leq 0.995 \)

with addition of the term:

\[
1.5 \left( |x - 1.25| \right)^3 - 0.025
\]

to the resulting \( x \) if the latter is in the range \( 1 \leq x \leq 2 \) (i.e. \( 0.84 \leq y \leq 0.005 \)). The percentage error in the calculated values compared with tabulated values is \( \pm 0.5\% \) which is acceptable in engineering calculations and only a very small price to pay for simplicity and speed of calculation.

REFERENCES


**FIGURE 1:** The grade efficiency curve $G(x)$ and the reduced grade efficiency curve $G'(x)$ for a hydrocyclone.

**FIGURE 2:**
Definition of the analytical cut size $x_a$.

**FIGURE 3:**
Definition of the intersection cut size $x_i$. 

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A 26
FIGURE 4: Plot of $G(x)$ against $F(x)$ for three different feed size distributions.
$F(x)$, showing the relative position of the three cut sizes $x_{50}$, $x_a$, and $x_L$ with respect to the feed median $x_g$.
Curve C - coarse feed, i.e. $x_g > x_{50}$
Curve B - $x_g = x_{50}$
Curve A - fine feed, i.e. $x_g < x_{50}$

FIGURE 5: A plot of Equation (12) with equal increments of $\frac{\ln g_s}{\ln g}$.
Example 8

Determine the number and diameter of hydrocyclones (Rietema's optimum separation design) for the following operating conditions:

\[
Ap = 305.24 \text{ kPa}, \ x_50 = 8 \mu m, \ \rho_s = 2640 \text{ kg m}^{-3}, \ \rho = 1000 \text{ kg m}^{-3},
\]
\[
\mu = 0.001 \text{ Ns m}^{-2}, \ \Omega = 8.333 \times 10^{-3} \text{ m}^{-3} \text{ s}^{-1}, \text{ feed solids concentration less than 1% by volume.}
\]

For Rietema's cyclone

\[
\Phi_{ke} \cdot \epsilon_u = 0.0611
\]

\[
\epsilon_u = 24.38, \ \frac{0.3748}{\epsilon_u} = \frac{\Omega}{Re}
\]

Ans. 2 cyclones, \( D_c = 91 \text{ mm} \)

Find the overflow concentration \% by volume of a hydrocyclone operated with feed concentration of 3% by volume if the reduced grade efficiency and the feed solids particle size distribution are as follows:

<table>
<thead>
<tr>
<th>Particle size (( \mu m ))</th>
<th>Feed solids (% undersize)</th>
<th>Reduced grade efficiency (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>5.7</td>
<td>4.0</td>
</tr>
<tr>
<td>5</td>
<td>18.8</td>
<td>20.1</td>
</tr>
<tr>
<td>7</td>
<td>35.9</td>
<td>40.6</td>
</tr>
<tr>
<td>10</td>
<td>56.0</td>
<td>65.5</td>
</tr>
<tr>
<td>15</td>
<td>76.9</td>
<td>86.9</td>
</tr>
<tr>
<td>20</td>
<td>87.5</td>
<td>94.9</td>
</tr>
<tr>
<td>30</td>
<td>95.9</td>
<td>99.1</td>
</tr>
</tbody>
</table>

\( N_u = 28\) \( \mu m = 136\% \)
3. Consider a separator operated in a multiple pass system by which the fresh feed at flowrate \( Q \) goes into a holding tank and the separator is supplied from this tank via a pump. One half of the separator overflow is re-cycled back into the holding tank and the other half is the final effluent from the system. Calculate the total mass recovery of the whole plant at a steady state continuous operation if the total efficiency of the separator is 47.87% and if the underflow flowrate is negligible.

Discuss the advantages and disadvantages of multiple pass arrangements and name a few types of separators suitable for such arrangements.

Determine the diameter and the minimum number of hydrocyclones to be operated in parallel to treat a slurry of the following properties:

- Liquid viscosity \( \mu \) = 0.001 Ns m\(^{-2}\)
- Liquid density \( \rho \) = 1000 kg m\(^{-3}\)
- Solids density \( \rho_s \) = 2600 kg m\(^{-3}\)
- Suspension flowrate \( Q \) = 0.01 m\(^3\) s\(^{-1}\)
- Pressure drop \( \Delta p \) = 10\(^5\) Nm\(^{-2}\)

The operating cut size is to be no more than 10 \( \mu m \). Assume that the feed solids concentration is low and the following relationships apply for scale-up.

\[ Stk_{50} \cdot Eu = 0.1111 \]

\[ Eu = 446.5 \times (Re)^{0.323} \]

where \( Stk_{50} \), \( Eu \) and \( Re \) are all based on cyclone diameter and the superficial velocity in the cyclone body.
Two hydrocyclones were used for the separation of solids from a given suspension. The suspension flow rate \( Q \) was the same in both cases and equal to 5 litres/sec., the pressure was also the same and the input concentration was 20\% by mass. The following results were obtained:

<table>
<thead>
<tr>
<th></th>
<th>Hydrocyclone 1</th>
<th>Hydrocyclone 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Underflow flow rate (l/s)</td>
<td>1.25</td>
<td>1.00</td>
</tr>
<tr>
<td>Solids concentration in</td>
<td></td>
<td></td>
</tr>
<tr>
<td>underflow (% by mass)</td>
<td>0.568</td>
<td>0.7</td>
</tr>
</tbody>
</table>

If a choice is to be made between the two hydrocyclones on the basis of their performance, which one would you find more efficient? What criterion have you used and why?

Discuss the main advantages and disadvantages of hydrocyclones in solid-liquid separation. What are their other uses? Sketch a typical hydrocyclone and the flow pattern within it: what causes the existence of an air column in the flow in some cases?

\[ A 30 \]
Appendix K-7

Performance of Hydrocyclones
At
High Feed Solids Concentration
PERFORMANCE OF HYDROCYCLONES AT HIGH FEED SOLIDS CONCENTRATIONS

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Summary

The effect of solids concentration on separation performance of hydrocyclones is well recognised in practice but very little quantitative information exists in the literature. A brief review of the current knowledge in the area is followed by a proposal of a semi-empirical dimensionless correlation which relates the reduced cut size to the operating variables including the solids concentration and the underflow-to-throughput ratio. The validity of the correlation is shown by comparison with the results of tests performed with 125 mm and 50 mm diameter hydrocyclones at varying feed concentrations.

The equation proposed here allows scale-up and predictions of performance in applications with feed concentrations higher than 8% by volume when the available theories cannot be applied.
NOMENCLATURE

$c$  Solids concentration by volume
$c_o$  Solids concentration in the feed
$c_u$  Solids concentration in the underflow
$D_C$  Diameter of hydrocyclone
$D_I$  Inlet diameter
$D_O$  Overflow diameter
$D_U$  Underflow diameter
$h_v$  Height of liquid surface
$h_{vz}$  Vortex finder length
$k_1, k_2$  Constants
$q_o$  Overflow rate
$q_u$  Underflow to throughput ratio
$q_f$  Feed flowrate
$q_u$  Underflow rate
$v_s$  Settling velocity
$v_{50}$  Constant (Eqn. 4)
$x_{50}$  Cut size
$A_p$  Total pressure drop
$\rho_{s}$  Density difference $= \rho_s - \rho_f$
$\rho_s$  Liquid viscosity
$\rho_s$  Density of solids
$\rho_f$  Density of liquid
$c_w$  Solids concentration by weight
1. INTRODUCTION

The feed concentration of solids is a very important variable which is known to affect both the separation efficiency and the pressure drop of a given hydrocyclone. It is however a relatively neglected effect in terms of literature information and little is known about it quantitatively. The purpose of this work was to construct a dimensionless correlation on the basis of some predictions in the literature and by analogy with hindered settling under gravity, and test its validity experimentally. The study is mostly concerned with correlation of the reduced cut size with the feed solids concentration but some notes on the effect on applied pressure drop are also given.

2. PREDICTIONS OF CUT SIZE AT LOW CONCENTRATIONS

The best way to characterise hydrocyclone separation efficiency is by a full grade efficiency curve. For correlation with different operating variables however preferably a single number is needed to characterise the performance. The cut size \( X_{50} \), i.e. the particle size corresponding to 50% on the grade efficiency curve is suitable and widely used for this purpose.

For a hydrocyclone of given configuration operated at low input concentrations and low underflow-to-throughput ratios, the cut size \( X_{50} \) may be assumed to depend only on cyclone diameter \( D_c \), liquid viscosity \( \mu \) and density \( \rho \), the difference between the density of the solids and the liquid \( \rho_s - \rho \) and suspension flowrate \( Q_s \). (Note that pressure drop \( AP \) is not listed because there is a fixed relationship between \( Q_s \) and \( AP \) so that only one of these can be taken into the analysis as an independent variable.)

Dimensional analysis shown in Reference 1 leads to a general relationship between three dimensionless groups. The actual form of the function has to be derived from the physics of the separation process. Despite the differences in the assumptions made by different authors, most of the theoretical correlations in the literature can be shown (Ref. 1) to lead to the form:

\[
X_{50} = K \left( \frac{\mu D_c}{Q_s \rho} \right)^{0.5}
\]

where \( K \) is a constant for a given cyclone design and it varies from 1.7 \( \times \) 10\(^{-2} \) to 2.3 \( \times \) 10\(^{-2} \). A closer study of some of the theories available reveals that \( K \) varies a little with Reynolds number but this can be neglected if \( Re \) does not change very much.

An important phenomenon to be observed from Equation (1) is that for a given pressure the cut size is proportional to \( D_c^{3/2} \) which means that higher efficiencies are obtained with smaller diameter cyclones. It therefore makes sense to use a number of smaller cyclones in parallel where maximum solids recovery is desirable. Unfortunately, most industrial applications of hydrocyclones are at higher feed solids concentrations and that is when difficulties arise because Equation (1) cannot then be applied.

There is no relationship at present which would give reliable prediction of cyclone performance under such circumstances. All we know is that, with increasing concentration, the separation efficiency goes down appreciably, i.e. the cut size increases. As at higher input concentrations the underflow to throughput ratio \( R_f \) has to be increased (to allow greater volume of separated solids to be discharged) by opening up the underflow orifice, the factor \( R_f \) also affects the separation efficiency. The control of the underflow orifice in fact plays an important part in the operation of hydrocyclones and for that purpose most cyclones are equipped with either a continuously variable underflow orifice (operated mechanically or pneumatically) or a series of replaceable nozzles. The dilemma is that a thick underflow and a high solids recovery cannot be achieved at the same time and one has to be sacrificed for the other. For maximum solids recoveries, the solids concentration in the underflow...
should be below a certain limit because above that limit some solids will start going into the overflow (Ref. 3). It means that this is at the expense of total separation efficiency. The practical limit of underflow concentrations achievable is about 50% by volume for materials like limestone or coal slurries. Some devices exist for automatic control of the underflow orifice in order to keep the underflow concentration below the value.

The effects of solids concentration on the performance of hydrocyclones is by far the most important phenomenon yet not satisfactorily quantified and explained. Presence of particles modifies the flow patterns in the cyclones and this combined with the inevitable hindered settling at high concentrations is the explanation for such effects.

3. CUT SIZE AT HIGH CONCENTRATIONS

The solid content of the feed has been found to be the variable which influenced the magnitude of cutsize the most. High solids concentrations have several effects on cyclone performance:

(i) hindered settling and thereby departure from Stokes' law;
(ii) an increase in the effective pulp viscosity;
(iii) even higher concentrations in the apex of the cone with resultant alterations of the character of the underflow stream;
(iv) change in pressure drop or capacity;
(v) change in flow pattern.

There are three aspects of the derivation of Stokes' law with respect to its applicability to separation with cyclones.

(a) It refers to streamline flow condition.
(b) It is a relationship for the unhindered movement of single particles.
(c) It applies to conditions under which forces are balanced, not to the period of particle acceleration prior to the attainment of balance.

The effects of concentration are considered to be negligible below a feed concentration which is equivalent to an 8:1 fluid to solid volume ratio (Ref. 4). This corresponds to 11 per cent v/v of solids: 25 per cent v/w for a solid density 2.7 g/cm³ in water. This would appear to be the high side since the concentration in the apex for a feed at 25 per cent v/w would be very high. Alternative limits quoted have been 15 per cent w/v (Ref. 5) and 8 per cent v/v. If the material is markedly non-spherical the suggested limit was reduced to 4 per cent v/w. Zhevnowatyi (Ref. 6) experimentally showed no effect up to a solid-liquid weight ratio of 0.3 with solid of 2.34 g/cm³ in liquid of density 1.34 g/cm³. This corresponds to quite a high figure of volumetric concentration of 17 per cent v/v. The limit is also probably dependent on cyclone diameter (Ref. 2).

Before embarking on analysis of literature for a useful correlation it should be pointed out here that, as at higher feed concentrations, the underflow-to-throughput ratio Xo is no longer negligible; it becomes another important dimensionless group likely to affect Xo. It not only affects the flow in the cyclone but also leads to a guaranteed efficiency because of the dead flux it causes. The latter effect is eliminated by using a modified definition of efficiency ("reduced" efficiency) from which a "reduced" cut size is derived. This is a widely accepted approach and the reader is referred to Reference 1 for further explanation. From now on Xo should be understood to mean the reduced cut size.

Qualitative information on the effect of feed concentration on the cut size is scarce. Leaving aside those sources that are either too vague or of very narrow applicability, there are two major empirical correlations reported recently.

Using 4, 6, 10 and 15 inch cyclones with slurries ranging from 15 to 70 per cent
solids by weight. Lynch (Ref. 7) developed the following equation:

\[
\log_{10} X_{50} = -0.04 + 0.0576 D_u - 0.0366 D_i + 0.0299 f - 0.0001 Q
\]  

(2)

where \( X_{50} \) is in microns

\( D_u, D_i, D_1 \) are diameters of the vortex finder, the underflow orifice and the

inlet respectively, in cm

\( f \) is \% w/w feed concentration

\( Q \) is in litres/min.

Pitt (Ref. 8) has suggested the following equation to show the effect of feed solids

content on the \( X_{50} \) cut size:

\[
X_{50} = \frac{-350 C_{0.6} 0.6 D_1 D_2 1.21 \exp(0.063c)}{D_u 0.71 h_{v_f} 0.38 0.45 (\rho_s - \rho_f)^{0.5}}
\]  

(3)

where \( h_{v_f} \) is the length of the vortex finder in inches

\( D_c, D_o, D_i \) and \( D_u \) are also in inches

\( \rho_s - \rho_f \) in g/ml

\( Q \) in cfs

\( X_{50} \) in \( \mu \)m, and

c is \% solid solids by volume.

Equations (2) and (3) proposed by two different workers both show an exponential

relationship between \( X_{50} \) and \( f \). Furthermore this ties in with a study of hindered

settling by Davies, Dollimore and McBridge (Ref. 9) who recently suggested the

following settling velocity-concentration relationship:

\[
V = V_s 10^{-bc}
\]  

(4)

where \( c \) is solid concentration volume

b is a constant and \( V_s \) is a constant.

This equation can be shown to be very closely related to the well-known Richardson-

Zaki equation for hindered settling (Ref. 9).

The above shown exponential relationship combined with Bradley's well tested

relation (Ref. 2, Eqn. (37)):

\[
X_{50} \sim (1 - R_T)^{0.5}
\]  

(5)

and with Equation (1) give the following equation first proposed by Svarovksy (Ref. 1):

\[
\frac{X_{50}}{D_c} = k_1 \left[ \frac{\nu D_c}{Q \Delta p} \right]^{\frac{1}{2}} (1 - R_T)^{\frac{1}{2}} \exp k_2 c
\]  

(6)

Here \( k_1 \) and \( k_2 \) are constants

c is the concentration of solids in the feed (v/v).
This semi-empirical equation has two advantages over Equations (2) and (3) which are purely empirical and established by regression analysis of experimental results. Firstly, it takes into account the effects of liquid viscosity, \( \mu \), and the underflow-to-throughput ratio \( R_f \) which Equations (2) and (3) do not. Secondly, for \( R_f = 0 \) and \( c = 0 \) it reduces into the well accepted Equation (1).

It does not, however, correlate \( X \) with the geometrical proportions of the hydrocyclone but this can be done easily by comparison with Equation (3). We consider this rather doubtful and prefer to establish the constant \( k_1 \) and perhaps also \( k_2 \) for different "standard" designs reported in the literature from tests.

The main purpose of the experimental work reported in the following was to examine the above correlation and test its validity.

4. DESCRIPTION OF THE TEST APPARATUS

The programme of test work was carried out on a test rig installed at the University of Bradford. Figure 1 shows the flow diagram of the rig. It consists of a 300 litre baffled tank and a weir box, which collects overflow from the two hydrocyclones (single and multiple), and overflows into the tank. The tank is kept about 2/3 full with a slurry of calcium carbonate. An agitator is used to keep the fine solids in suspension. The agitation created by recirculation of the slurry also helps the conditions within the tank to be nearly homogeneous. The feed is pumped through the hydrocyclones using centrifugal pumps.

4.1 Single Hydrocyclone

This hydrocyclone, model HW 2515, is manufactured by AKW of West Germany. The whole cyclone is made from Vulkolan (a polymer based on polyurethane), the underflow nozzle is simply pushed inside the cyclone apex and secured by a jubilee cap.

The dimensions of the hydrocyclone are:

- Length = 78 cm
- Cylindrical section length = 28 cm
- Cylindrical section diameter = 12.5 cm
- Vortex finder diameter = 4 cm
- Underflow orifice diameter = 1.6 cm
- Feed inlet diameter = 2.52 cm
- Angle of cone = 15°

The maximum feed pressure employed is 15 psi because the bucket collecting the overflow stream from the hydrocyclone would otherwise overflow at higher feed pressures.

4.2 Multi-Hydrocyclone

The multi-hydrocyclone is that of Moxley 2" assembly. The assembly consists of 4 Moxley hydrocyclones of diameter 2", placed in parallel. All the hydrocyclones are identical and each hydrocyclone is of the following dimensions:

- Length = 38 cm
- Cylindrical section diameter = 5.08 cm
- Feed inlet size (rectangular) = 8 mm x 6 mm
- Angle of cone = 6°
- Underflow orifice sizes = 3/16", 1/4", 5/16"
- Vortex finder sizes = 5/16", 7/16", 9/16"
With the multi-cyclone it was possible to vary the operating pressure up to 30 psi.

Operation of the Test Rig

The 300-litre vessel is filled up to the 250-litre mark with water. The required amount of powder is added and the agitator is switched on. Teepol is added dropwise until a noticeable amount of froth starts to form. Teepol acts as a surfactant and helps to disperse the particles. Samples of fluid are taken before and after adding powder. The appropriate valves (1, 4 and 5 in the case of the multi-hydrocyclones) are opened and the pump is switched on. The slurry is now being pumped back into the tank. Valve number 6 is slowly opened and valve number 5 is closed. The feed pressure and the flowrate can be controlled by adjusting valve number 6. Each hydrocyclone has a pressure indicator connected to it. Upon entry, the inlet slurry is split into two streams. The fine product stream leaves the hydrocyclone through the vortex finder and falls into a bucket. This then runs into the weir box and finally overflows into the feed tank. The coarse product leaves through the underflow orifice and flows back into the tank.

The unit is allowed to run for some time so that steady-state is reached before taking the readings and samples. Then feed pressure, temperature of the slurry, the height of the liquid level in weir box are noted. The underflow rate is measured using a bucket and a stopwatch. Samples from underflow and overflow are taken to determine concentration and particle size distribution. The same procedure is repeated with the single hydrocyclone before increasing the concentration of the feed slurry to the next required level.

4.4 Procedure to Increase the Concentration of the Feed Slurry

Before increasing the slurry concentration the existing concentration of the feed has to be determined. With the help of the data obtained in the previous run, the feed concentration is estimated using the following formula. (Note that estimates of \( c_f \) by taking samples from the feed tank are highly inaccurate due to particle stratification in the vessel.)

\[
\frac{c_f}{c_o} = \frac{U_f c_o + O_f c_o}{U_f + O_f}
\]

where
- \( c_f \) = concentration of feed
- \( c_o \) = concentration of underflow
- \( c_o \) = concentration of overflow
- \( U_f \) = underflow rate
- \( O_f \) = overflow rate

The volume of the slurry present in the cylindrical feed tank is determined by measuring the height of the fluid and the diameter of the tank. Once the concentration of the slurry is known, the amount of solids to be added in order to achieve required concentration is calculated.

4.5 Measurement Procedure

4.5.1 Underflow rate

The underflow rate is determined by measuring the amount collected in a bucket for a certain length of time. (Typically 15 secs.) Several readings are taken and the average value is taken to reduce errors.

4.5.2 Overflow rate

The overflow rate is measured by using the V-notch type weir box.

The flow through such a weir box is given by the following equation obtained by
calibration:

\[ Q = 0.882 (H - 0.94)^{2.55} \]  \hspace{1cm} (8)

where \( H \) is height of the liquid surface above the bottom of the weir box in m and \( Q \) is flowrate in m/s.

4.5.3 Feed flowrate

The feed flowrate is obtained by adding the underflow and the overflow rates.

4.5.4 Determination of concentrations

A known volume of (25 cc) slurry is pipetted from underflow or overflow and taken to a pre-weighed beaker. The sample is left in a dryer until it is completely dry. The difference of the initial and final weight of the beaker gives the weight of powder present in 25 cc of slurry. Knowing the density of powder and that of the medium (water) it is possible to calculate the volumetric concentration (% v/v).

4.5.5 Particle size distribution

In order to draw the grade efficiency curve, it is necessary to know the particle size distribution of underflow and overflow of each run. This is done using the Andreasen pipette method.

4.5.6 Feed material

Finely ground limestone was used as feed material. The limestone was found to be suitable because of its better dispersion in water. The density of the material is 2.71 g/cm³. The particle size analysis of the feed by the Andreasen pipette method in water is shown in Figure 2.

5. RESULTS AND DISCUSSION

During the course of these tests, the feed concentration was gradually increased (from 3 v/v % to 28 v/v %) keeping all other operational variables and sizes of nozzles constant. The results are reported here in tabular form. Tables 1 and 2 give the measured and calculated operational variables while Tables 3 and 4 give the final reduced cut sizes derived from grade efficiency curves (Ref. 10).

TABLE 1
A Summary of Test Results

(single hydrocyclones - feed conc. 3 v/v % - 27 v/v %)

<table>
<thead>
<tr>
<th>Exp No.</th>
<th>Vortex Finder Size (mm)</th>
<th>Under Flow Orifice Size (mm)</th>
<th>Pressure Drop psi</th>
<th>Temp of Feed C</th>
<th>Over Flow Rate Litre/s</th>
<th>Under Flow Rate Litre/s</th>
<th>Total Flow Rate Litre/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>40</td>
<td>16</td>
<td>15</td>
<td>19.0</td>
<td>3.140</td>
<td>0.46</td>
<td>3.600</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td></td>
<td>28.0</td>
<td>3.357</td>
<td>0.59</td>
<td>3.947</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
<td></td>
<td>22.0</td>
<td>3.360</td>
<td>0.46</td>
<td>3.820</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
<td></td>
<td>28.0</td>
<td>3.079</td>
<td>0.22</td>
<td>3.299</td>
</tr>
<tr>
<td>5</td>
<td></td>
<td></td>
<td></td>
<td>25.0</td>
<td>3.079</td>
<td>0.21</td>
<td>3.289</td>
</tr>
<tr>
<td>6</td>
<td></td>
<td></td>
<td></td>
<td>25.0</td>
<td>3.079</td>
<td>0.22</td>
<td>3.299</td>
</tr>
<tr>
<td>7</td>
<td></td>
<td></td>
<td></td>
<td>26.0</td>
<td>3.079</td>
<td>0.23</td>
<td>3.310</td>
</tr>
</tbody>
</table>
**TABLE 1 Continued**

<table>
<thead>
<tr>
<th>Exp No.</th>
<th>Under Flow Conc. g/l</th>
<th>Overflow Conc. g/l</th>
<th>Feed Conc. g/l</th>
<th>Under Flow Conc. %</th>
<th>Feed Conc. % v/v</th>
<th>Under Flow to Thruput Ratio ( R_f )</th>
<th>Total Efficiency %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>105.9</td>
<td>78.0</td>
<td>81.5</td>
<td>4.1</td>
<td>3.1</td>
<td>0.166</td>
<td>12.8</td>
</tr>
<tr>
<td>2</td>
<td>165.9</td>
<td>83.4</td>
<td>95.8</td>
<td>6.5</td>
<td>3.7</td>
<td>0.260</td>
<td>15.1</td>
</tr>
<tr>
<td>3</td>
<td>241.0</td>
<td>262.9</td>
<td>316.0</td>
<td>19.4</td>
<td>13.2</td>
<td>0.168</td>
<td>12.0</td>
</tr>
<tr>
<td>4</td>
<td>212.9</td>
<td>398.1</td>
<td>418.2</td>
<td>35.5</td>
<td>18.2</td>
<td>0.064</td>
<td>10.8</td>
</tr>
<tr>
<td>5</td>
<td>246.5</td>
<td>475.8</td>
<td>492.7</td>
<td>37.6</td>
<td>22.0</td>
<td>0.064</td>
<td>9.6</td>
</tr>
<tr>
<td>6</td>
<td>233.0</td>
<td>520.7</td>
<td>536.2</td>
<td>38.5</td>
<td>24.6</td>
<td>0.067</td>
<td>9.4</td>
</tr>
<tr>
<td>7</td>
<td>261.6</td>
<td>573.3</td>
<td>586.5</td>
<td>39.0</td>
<td>27.5</td>
<td>0.069</td>
<td>9.1</td>
</tr>
</tbody>
</table>

**TABLE 2**

A Summary of Test Results

(multiphycyclones - feed conc. 3 % 9/10 - 27 % 11)

<table>
<thead>
<tr>
<th>Exp No.</th>
<th>Vortex Finder Size (in)</th>
<th>Under Flow Orifice Size (in)</th>
<th>Pressure Drop psi</th>
<th>Temp of Feed °C</th>
<th>Over Flow Rate L/S</th>
<th>Under Flow Rate L/S</th>
<th>Total Flow Rate L/S</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>9/16</td>
<td>3/16</td>
<td>28</td>
<td>20.0</td>
<td>2.351</td>
<td>0.140</td>
<td>2.491</td>
</tr>
<tr>
<td>9</td>
<td></td>
<td>28</td>
<td>25.5</td>
<td>2.473</td>
<td>0.130</td>
<td>2.603</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td></td>
<td>29</td>
<td>24.0</td>
<td>3.210</td>
<td>0.143</td>
<td>3.353</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td></td>
<td>30</td>
<td>24.0</td>
<td>2.412</td>
<td>0.140</td>
<td>2.552</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td></td>
<td>-</td>
<td>23.0</td>
<td>2.412</td>
<td>0.140</td>
<td>2.552</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td></td>
<td>-</td>
<td>20.0</td>
<td>2.455</td>
<td>0.200</td>
<td>2.745</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td></td>
<td>-</td>
<td>22.0</td>
<td>3.445</td>
<td>0.200</td>
<td>3.644</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td></td>
<td>-</td>
<td>24.0</td>
<td>2.070</td>
<td>0.184</td>
<td>2.254</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Exp No.</th>
<th>Under Flow Conc. g/l</th>
<th>Over Flow Conc. g/l</th>
<th>Feed Conc. g/l</th>
<th>Under Flow Conc. %</th>
<th>Feed Conc. % v/v</th>
<th>Under Flow to Thruput Ratio</th>
<th>Total Eff. %</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>386.6</td>
<td>75.6</td>
<td>93.1</td>
<td>16.6</td>
<td>3.7</td>
<td>0.056</td>
<td>23.3</td>
</tr>
<tr>
<td>9</td>
<td>556.7</td>
<td>132.0</td>
<td>153.1</td>
<td>25.8</td>
<td>6.0</td>
<td>0.048</td>
<td>17.5</td>
</tr>
<tr>
<td>10</td>
<td>332.1</td>
<td>176.3</td>
<td>204.3</td>
<td>44.3</td>
<td>8.2</td>
<td>0.043</td>
<td>17.4</td>
</tr>
<tr>
<td>11</td>
<td>246.9</td>
<td>182.6</td>
<td>218.0</td>
<td>43.9</td>
<td>8.8</td>
<td>0.055</td>
<td>20.8</td>
</tr>
<tr>
<td>12</td>
<td>271.5</td>
<td>247.8</td>
<td>282.0</td>
<td>47.4</td>
<td>11.6</td>
<td>0.055</td>
<td>17.0</td>
</tr>
<tr>
<td>13</td>
<td>386.3</td>
<td>347.7</td>
<td>386.9</td>
<td>48.1</td>
<td>16.6</td>
<td>0.073</td>
<td>16.7</td>
</tr>
<tr>
<td>14</td>
<td>949.2</td>
<td>464.2</td>
<td>491.2</td>
<td>51.9</td>
<td>22.1</td>
<td>0.054</td>
<td>10.6</td>
</tr>
<tr>
<td>15</td>
<td>1010.0</td>
<td>528.9</td>
<td>568.2</td>
<td>59.3</td>
<td>26.5</td>
<td>0.082</td>
<td>14.5</td>
</tr>
</tbody>
</table>
TABLE 3
Values of Cut Size Against Feed Concentration
(single hydrocyclone)

<table>
<thead>
<tr>
<th>Experimental No.</th>
<th>Feed Conc. v/v %</th>
<th>Reduced Cut Size $X_{50}$ μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3.1</td>
<td>11.2</td>
</tr>
<tr>
<td>2</td>
<td>3.7</td>
<td>15.7</td>
</tr>
<tr>
<td>3</td>
<td>13.2</td>
<td>26.8</td>
</tr>
<tr>
<td>4</td>
<td>18.2</td>
<td>36.5</td>
</tr>
<tr>
<td>5</td>
<td>22.0</td>
<td>39.0</td>
</tr>
<tr>
<td>6</td>
<td>24.7</td>
<td>42.0</td>
</tr>
<tr>
<td>7</td>
<td>27.5</td>
<td>47.0</td>
</tr>
</tbody>
</table>

TABLE 4
Values of Cut Size Against Feed Concentration
(multi hydrocyclone)

<table>
<thead>
<tr>
<th>Experimental No.</th>
<th>Feed Conc. v/v %</th>
<th>Reduced Cut Size $X_{50}$ μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>3.7</td>
<td>10.4</td>
</tr>
<tr>
<td>9</td>
<td>8.0</td>
<td>14.4</td>
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<td>10</td>
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<td>18.5</td>
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<td>12</td>
<td>11.6</td>
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<td>13</td>
<td>16.6</td>
<td>20.5</td>
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<td>14</td>
<td>22.1</td>
<td>23.0</td>
</tr>
<tr>
<td>15</td>
<td>26.5</td>
<td>29.5</td>
</tr>
</tbody>
</table>

Comparisons of experimental data with Svarovsky's correlation in Equation (6) (Figures 3 and 4) show that at low feed concentrations there are appreciable differences between experimental values with those predicted by the correlation. However, at higher concentrations (above 8 v/v %) there is a good agreement and the maximum differences are 4.53% and 7.19% for single and multi-hydrocyclones respectively. A comparison of the values of constants estimated on the basis of experimental data with those, obtained by Svarovsky and Ravishi (Ref. 3) is given in Table 5.

Apart from cyclone configuration and diameter, the material of feed may also be a factor which affects these constants.

With respect to the effect of feed concentration on pressure drop this was also studied but detailed results are not given here and can be found in Reference 10. As found by many other workers before, the pressure loss factor showed an initial decrease with increasing concentration and after going through a minimum at 8% v/v for the single cyclone and 12% for the multi-cyclone it went up again. The initial decrease is usually attributed to suppression of turbulence and changes in flow patterns.
caused by the presence of particles while the latter increase at high solids concentrations is due to a dramatic increase in the effective slurry viscosity.

6. CONCLUSIONS

The correlation proposed in Equation (6) holds reasonably well for a wide range of feed concentrations (8 v/v % to 28 v/v %) and the values of constants in the correlation may depend on cyclone configuration, cyclone diameter and on the feed solids.

Although two different cyclone sizes were used in this study the effect of cyclone diameter could not be shown because the cyclones were of different geometrical proportions.

Limestone being the only material used in this work, it was not possible to examine the effect of the feed material and its properties but, in analogy with hindered settling under gravity, it is very likely that this effect is appreciable. Further work is now necessary to establish the above mentioned relationships so that the proposed equation can be used in scale-up.

Similarly to hindered settling under gravity, scale-up of hydrocyclones operated at high solids concentrations has to be based on laboratory or pilot type test work on geometrically similar cyclones because the particle-fluid and particle-particle interactions inside the hydrocyclones are extremely complex.

The pressure loss of a hydrocyclone is influenced by the concentration of feed. Although initially there is a decrease in pressure loss along with the increase in feed concentration which is due to suppression of turbulence and changes in flow profiles by the presence of particles, at higher concentrations pressure loss increases with increase in feed concentration when the effect due to the increase in effective viscosity becomes predominant.

7. ACKNOWLEDGEMENTS

We are very grateful to the Queen's Gate Whiting Company, Beverly, Yorkshire, for the limestone used in the tests and to Richard Mosley Ltd., Falmouth, Cornwall for letting us have their multi-cyclone unit.
8. REFERENCES


PI Pressure Indicator

Multi-Hydrocyclone

Bucket

Single Hydrocyclone

Weir Box

Baffled Tank (300 litres)

Centrifugal Pumps

Flow Meter

[Key: Numbers 1 - 9 Indicate Valves]

**Fig. 1** A Flow Diagram of the Test Rig
FIGURE 2: Particle size distribution of the feed
FIGURE 3: COMPARISON OF EQUATION (6) WITH EXPERIMENTAL DATA OBTAINED BY USING SINGLE HYDROCYCLONE
Figure 4: Comparison of Equation (6) with experimental data obtained by using multi-hydrocyclone.
either a continuously variable underflow orifices (operated
or a series of replaceable nozzles. The dilemma is that a thick underflow and a high
solids recovery cannot be achieved at the same time and one has to be sacrificed for
the other. For maximum solids recoveries, the solids concentration in the underflow

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