REMOVAL OF TOXIC ORGANICS FROM WASTEWATER BY ADSORPTION WITH GRANULAR ACTIVATED CARBON

by

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SUMMARY

1. A recently completed adsorption study indicates that most of the EPA proposed dissolved organic toxic chemicals can be removed from water by activated carbon (Benzidine was not tested).

2. Other chemically similar (aromatic, non-polar, high molecular weight) contaminants, such as OSHA defined carcinogens and the chemicals under examination by the EPA for inclusion on the toxic chemicals list are also predicted to be adsorbable from wastewater by activated carbon.

3. Full-scale granular activated carbon installations are currently removing toxic or biologically refractive contaminants from wastewater, and the exhausted carbon is being reactivated for reuse.

MONS 064557

WASTEWATER TREATMENT FOR TOXIC ORGANIC CHEMICALS REMOVAL WITH GRANULAR ACTIVATED CARBON

Prologue

During the early 1900's, a missionary traveling through Southeast Asia came upon a village with a very unique marital situation — every married man had nine wives. Being a world traveler, the missionary was not surprised by polygamy, but he did wonder about the number nine. Finally, when his curiosity got the best of him, he mustered enough courage to ask one of the tribesmen the purpose of nine wives.

"It is very simple," replied the man. "Our nearest clean water supply is nine miles away, and it requires one person a whole day to walk the distance both ways. It also takes nine trips to secure enough water to satisfy the needs of a family for one day. Since water-carrying duties are performed by our women, it becomes a practical matter to have nine wives!"

Introduction

Through the ages man has shown that he will adapt his social habits to the quest for clean water. Civilizations such as Egyptians, Romans, Greeks, and yes, even our early colonists, took care to protect and cleanse water supplies. And many of these peoples used charcoal to purify both water and medicines.

Although the adsorption process was known in ancient times, it was not until the 20th century that we began to use powdered carbon on a grand scale for the removal of taste- and odor-causing constituents from drinking water. Then, during World War I, powdered carbon was tried in gas masks to protect troops from toxic vapors used in chemical warfare. As powder, however, the carbon was not practical because the product was dusty, so granular carbons were developed.

Following World War II, carbon adsorption found applic[illegible] in sugar decolorization, solvent recovery, and liquid and gas purification. Later, the adsorption process was shown to be technically sound and economically attractive for the removal of refractory and toxic dissolved organics from waste streams.

Adsorption systems have been proven to be more efficient due to their ability to handle toxic wastes such as insecticides, herbicides, other chlorinated hydrocarbons and phenol. Scientists in many countries including Russia,[1,2] Switzerland[3] and the United States,[4,5,6,7] agree on this point, and their work is cited in the literature.

An example of the use of an adsorption system to correct a health hazard was experienced in Ohio in 1971 when a 5-acre state park lake was deliberately poisoned with 1.8 pounds of endrin[8]. To eliminate the hazard posed by the lake, both to human and aquatic life, the entire body of water was pumped through an adsorber containing granular carbon. Analyses showed the contaminated water contained up to 10 ppb endrin, while the carbon-treated effluent showed the endrin content to be below detectable limits. The spent carbon was thermally reactivated, and the endrin destroyed.

In another case, a railway spill of 25,000 gallons of carbolic acid threatened the Potomac River. Again, activated carbon was employed to remove the compound from the runoff entering a feeder creek, thereby protecting drinking water supplies downstream. And, during another emergency caused by an accidental spill of acrylonitrile in Ohio, carbon was employed in a potable water treatment plant to prevent the possibility of the chemical entering the drinking supply.

Although these examples illustrate the utility of carbon adsorption in specific instances, the questions posed today are: Which toxic organic chemicals give us immediate concern? Why? And can granular activated carbon remove them from our waste streams?

The Federal Water Pollution Control Act requires that EPA publish a list of toxic pollutants and establish effluent standards for such pollutants. The statute specifically provides that the standard can be an absolute prohibition of discharge. The list can be added to whenever the EPA finds other pollutants that meet the statutory criteria of "toxic pollutants." Thus far, the EPA has proposed effluent guidelines for the following toxic pollutants: aldrin, dieldrin, benzidine and its salts, cadmium and all cadmium compounds, cyanide and all cyanide compounds, DDD, DDE and DDT, endrin, mercury and all mercury compounds, polychlorinated biphenyls and toxaphene. With the exception of mercury, cadmium and cyanide, these chemicals are organic in nature and have cyclic structures (Figure 1). Additional toxic pollutant lists published in the future may include those shown in Figure 2.[9]

In addition to these lists, the administrators of the Occupational and Safety Health Act (OSHA) have listed 14 compounds as carcinogenic to humans or animals (Table 1).[10] These compounds also have cyclic structures and are similar to those appearing on the EPA list (Figure 3).

According to Irma West of the California State Department of Health, the long range or indirect effects that toxic organic chemicals can have on the environment have been most disturbing. The most widely used pesticides, the chlorinated hydrocarbons, are concentrated by living matter in the aquatic environment. An increasing buildup of the pesticide may occur in each link of the food chain. However, the amounts tolerated before noticeable effects occur vary with the particular animal species and the posticide. Some species can tolerate larger amounts, but sooner or later, they are affected by decreased fertility and eventually death.[11]

Although most attention to this type of buildup is focused on lower forms of animals such as shellfish and other marine life, it is a fact that DDT and its metabolate DDE have been found in all samples of human fat tested for these chemicals (Table 2).[12]

Much research is being conducted to determine the effect of toxic organic buildup in the human body. However, the complex nature of the task will undoubtedly require many years to develop clear-cut conclusions. On the other hand, we do know that many of these chemicals attack the central nervous system and liver when taken in large amounts.[12] And, as little as 0.04 ounces (20 mg/kg) of a chemical such as dieldrin can be considered a large amount when we take into account that this dose can kill a 170-pound man.[13]

To eliminate the threat of toxic organic chemicals as a potential health hazard then, we must take precautionary measures to keep them under control. And, as mentioned previously, the use of granular carbon can help to achieve that goal.

Adsorption Isotherm Study

The technical literature includes numerous laboratory studies regarding the viability of adsorption for removal of many of the EPA proposed toxic chemicals from wastewater — plus many other potential contaminants used in commerce for pesticides and organic chemical intermediates applications. These researchers have examined a variety of types of activated carbon under many water conditions. The results are invariably the same. These compounds are removed from water very effectively by adsorption in the presence of other organic chemicals. This is not surprising as carbon adsorption is favored by higher molecular weight, non-polarity and limited solubility of the contaminants, most of which characteristics the proposed toxic chemicals possess.

A laboratory isotherm study was recently completed to evaluate the removal of organic chemicals on the EPA list of toxic pollutants by a commercial-grade of granular activated carbon which has had considerable use in the U.S., Japan and Europe in numerous municipal and industrial wastewater treatment systems. The laboratory procedures were standard adsorption isotherm techniques described in the literature and specifically delineated in Addendum 1 and Addendum 2. Analytical testing for the concentration of contaminants followed EPA recommended procedures.[14]

A laboratory adsorption isotherm testing procedure is a reliable technique to ascertain feasibility of adsorption treatment. This testing procedure specifically indicates:

1. The effluent levels of contaminant concentration obtainable by adsorption treatment.

2. The weight of contaminant that will be adsorbed by the adsorbent at the contaminant concentrations studies.

On the other hand, the laboratory test is not used to determine the necessary contact time for granular carbon beds to effect the desired reduction. This determination is usually performed in small pilot carbon beds under dynamic hydraulic flow conditions — also, a well-established procedure reported in the literature.

Since the solubility in water of the proposed toxic chemicals is limited,[15,16] it is critical to prepare the standard isotherm test samples by filtration through a fine filter to remove the insoluble fraction (Addendum 1 and 2).

Data on removal of DDD, DDT, DDE, endrin, aldrin, dieldrin, PCB's (Arochlor 1242 and 1254), and toxaphene is shown in summary form in Figures 4, 5, 6 and 7. Benzidine is not shown because its inclusion on the OSHA list of carcinogens prevented its inclusion in this study.

MONS 064562

In each instance, the fine filtration step ahead of adsorption substantially reduced the concentration of contaminants from the initial 100 ppb levels. In the tests on the PCB mixtures (Figure 6), the samples indicated a high degree of insolubility, and thus filtration alone reduces the initial concentrations to levels proposed by the EPA guidelines. Other isomers of the PCB family may be less insoluble, and adsorption appears to effectively reduce the soluble fraction to very low concentrations.

Many of the isotherm results plotted in Figures 4-7 do not reach the lower effluent limits proposed in the effluent guidelines because of difficulties in testing techniques associated with these very low concentrations. However, isotherm data can be reasonably extrapolated beyond the data provided by the analytical techniques. Based on the data shown in Figures 4-6, experience with granular carbon systems indicates that the carbon adsorption process can be designed to meet treatment objectives proposed by the EPA.

Isotherm test data for toxaphene is not entirely conclusive, and the tests should be repeated to eliminate interferences and to assure the use of optimum adsorption conditions. Toxaphene is reported by Baron to be adsorbable on activated carbon.[5]

The data shown in Figures 4-7 and in Addendum 1 and 2 suggest the following conclusions of this adsorption isotherm study:

1. Adsorption on activated carbon is an effective treatment process to meet proposed EPA effluent objectives for toxic organic chemicals.
2. Weight pickup of the various contaminants at the very low concentrations tested varied between 0.5 and 10% by weight of carbon.
3. Fine media filtration removes substantial amounts of insoluble components in commercial-grade chemicals found on the proposed toxic chemicals list.
4. Analytical testing of the wastewater will require exacting procedures and techniques to assure meaningful results on the carbon-treated wastewater.

MONS 064563

The results of this isotherm study are consistent with a broad-gauge isotherm study recently completed on 222 samples of industrial effluents in which carbon adsorption was shown to be effective for 76% of the wastewaters tested.[17]

Full-Scale Design Parameters

The adsorption isotherm study previously described provides only a cursory view of adsorption's potential for removing toxic organic chemicals from waste streams. However, numerous companies have proceeded far beyond an isotherm test procedure to the installation of full-scale systems. Some have been in operation for a number of years, while others have more recently been put into service.

Each system is comprised of three basic functional components:

1. The adsorption treatment of the wastewater
2. The carbon reactivation equipment
3. The carbon/water transport arrangement

Adsorption Treatment of the Wastewater

The adsorbers hold the granular activated carbon beds through which the wastewater flows. They can be designed for pressure or gravity flow to achieve the desired contact time of the water with the carbon. Suspended solids and space considerations also must be considered in the adsorber configuration. Flow rates usually fall under 10 gpm per square foot of carbon bed surface area. Contact times for industrial wastewater mixtures usually are in excess of 60 minutes, which is about twice the time employed for purification of domestic sewage. When suspended solids are present, they will be filtered out on the carbon bed. This dual purpose of carbon beds can be usefully employed as long as the adsorbers are designed to accommodate periodic backwashing and bed-cleaning procedures, such as air scour and/or surface wash.

A well-designed water distribution or underdrain will insure good backwashing performances as well as an even distribution of the water. Well

established filtration design practices can be effectively employed in carbon bed design systems. Keep in mind, however, that the carbon beds must be periodically removed via water slurry and obstructions to the flow of carbon from the adsorber should be avoided in adsorber design.

Carbon beds are normally in excess of 10 feet deep and usually fall into one of four basic configurations:

1. Moving beds
2. Beds in series
3. Beds in parallel
4. Expanded beds

The type of configuration selected depends on a number of variables — principal among them are — total water flow and suspended solids and degree of contaminant reduction desired. These and other factors have been discussed in other papers on this subject.[17,18]

Reactivation of the Granular Carbon

Thermal oxidation is employed to reactivate the exhausted carbon for reuse. Either multiple hearth furnaces or rotary kilns are employed for reactivation of the exhausted carbon. The size of the thermal reactivation equipment is based on the carbon exhaustion rate, i.e., pounds of carbon exhausted per thousand gallons of wastewater treated, and the weight of contaminant on the carbon. Excess capacity is designed into the thermal reactivation unit to allow for variances in carbon use rate due to changes in the wastewater flow and organic loading.

The exhausted granular activated carbon is heated to 1600-1800°F to effect volatilization and oxidation of the dissolved organic contaminants. Oxygen in the furnace is normally controlled at less than 1% to effect selective oxidation of contaminants over activated carbon. A 5% loss of activated carbon per reactivation cycle is an acceptable bench mark upon which granular

carbon system economics may be based. In addition, the reactivation equipment should include an afterburner and/or air scrubber to reduce the possibility of air pollution.

Granular carbon is transported between the adsorbers and the reactivation equipment by water slurry. Various pumping designs can be employed including centrifugal and diaphragm pumps as well as movement using either hydraulic or pneumatic pressure. The transport piping should include flush ports and wide radius bends. About one to three pounds of carbon per gallon of water are used, depending upon distance and elevation considerations.

Materials of Construction

Special consideration should be given to the selection of materials of construction used in the adsorption system from two standpoints:

1. Galvanic Corrosions — any tendency toward galvanic corrosion due to water characteristics, such as conductivity and pH, will be enhanced in granular carbon beds. Tanks or adsorbers holding granular carbon under water made from mild steel should be lined or constructed of cement or reinforced synthetic resins/plastics.

   Carbon/water slurry piping, which experiences only periodic exposure, is usually exempted from any special corrosion considerations.

2. Erosion — periodic replacement of the carbon beds can cause lining failures at exit ports in carbon tanks and adsorbers. Special grades of stainless steel are usually employed for such wear points.

FULL-SCALE INDUSTRIAL INSTALLATIONS

Tables 3 and 4 summarize data from industrial granular activated carbon systems purifying wastewater that is toxic or biologically refractive. Table 3 includes those installations which include on-site thermal reactivation, while Table 4 summarizes data from installations where the carbon is transported to central reactivation facilities. The data is, of necessity, not detailed because most industrial dischargers classify such information as confidential.

The flow rates shown in Tables 3 and 4 vary considerably because of the point of application at each plant. In some cases, the total plant effluent is being treated. In others, only specific waste streams are being treated by adsorption. The latter point of application normally is beneficial from a total cost viewpoint, as there is usually less contaminated water (smaller treatment system) and higher contaminant concentrations (greater use of the carbon before reactivation).

Comparison of Table 3 and Table 4 on total costs indicates similar costs per 1000 gallons regardless of the carbon reactivation system being on-site or reactivation being performed on a service basis. While the total annual costs appear to be similar, there would be appreciably less initial investment at each treatment site and less space requirements if the reactivation system can be obviated by a reactivation service.

Total annual costs shown in Table 3 range from $1 to $20 per 1000 gallons. Similarly, total costs in Table 4 range from $1.15 to $11 per 1000 gallons. These ranges are clearly in excess of costs normally associated with domestic sewage or biodegradable industrial wastewater treatment. This can be attributed not only to the type and severity of the contaminant problems being handled

by carbon, but also due to the fact that there are no additional costs associated with granular carbon treatment. Biological treatment costs estimates often reflect only the wastewater treatment components while neglecting the associated sludge treatment, handling and disposal.

MONS 064568

CONCLUSION

Adsorption by activated carbon is an effective method to reduce EPA proposed toxic chemicals to EPA proposed discharge guidelines. A number of full-scale granular carbon installations have demonstrated reliable and economic treatment of wastewaters containing toxic or biologically refractive contaminants. Granular carbon treatment has been employed on total plant effluents as well as specific in-plant waste streams. Granular carbon reactivation can be performed economically at the treatment site or central reactivation facilities on a service basis.

MONS 064569

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and

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MONS 064570

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MONS 064571

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MONS 064572

TABLE 1

LIST OF CARCINOGENS

2-Acetylaminofluorene

4-Aminodiphenyl

Benzidine (and its salts)

bis-Chloromethyl ether

3,3'-Dichlorobenzidine (and its salts)

4-Dimethylaminoazobenzene

Ethyleneimine

Methyl Chloromethyl ether

4,4'-Methylene(bis)-2-chloroaniline

alpha-Napthylamine

beta-Napthylamine

4-Nitrobiphenyl

N-Nitroso dimethylamine

beta-Propiolactone

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TABLE 2

DDT PLUS DDE IN HUMAN TISSUE (mg)

AGE	SEX	BLOOD	BRAIN	KIDNEY	LIVER	PROTEIN	FAT	TOTAL
48	M	0.230	0.30	0.005	2.0	4.0	290	300
40	M	0.079	0.06	0.05	1.0	2.0	69	72
41	F	0.053	0.04	0.03	0.2	0.4	41	42
76	F	0.012	0.02	0.02	0.2	0.7	21	22

MONS 064574

TABLE 3

TOXIC OR REFRACTORY INDUSTRIAL WASTEWATER TREATMENT
GRANULAR CARBON INSTALLATIONS (ON-SITE REACTIVATION)

(Partial Listing)

PLANT	FLOW MILLION GAL/YEAR	TOXIC OR REFRACTORY CONTAMINANTS AVERAGE INFLUENT	EFFLUENT OBJECTIVE	TYPE OF THERMAL ON-SITE REACTIVATION	CAPITAL COSTS* ($000)	DIRECT OPERATING COSTS 1000 GALLONS**
A[19]	6	10,000 mg/l TOC	400 mg/l TOC	Multiple Hearth	350	$20
B[20]	180	370 mg/l phenolics	10 mg/l phenolics	Multiple Hearth	1,300	2
C	70	300 mg/l phenolics	1 mg/l phenolics	Kiln	450	1
D[21]	50	100 mg/l phenolics	1 mg/l phenolics	Multiple Hearth	300	4
E	550	Color	Color removal	Kiln	---	---
F	1100	Color	Color removal	Multiple Hearth	---	---

* = Includes engineering, construction, adsorption system and reactivation equipment

** = Direct operating costs and do not include depreciation or cost of capital invested.

MONS 064575

TABLE 4

TOXIC OR REFRACTORY INDUSTRIAL WASTEWATER TREATMENT
GRANULAR CARBON INSTALLATIONS (CENTRAL REACTIVATION)

PLANT	FLOW MILLION GALLONS PER YEAR	TOXIC OR REFRACTORY CONTAMINANTS AVERAGE INFLUENT		EFFLUENT OBJECTIVE	TOTAL ANNUAL COSTS $/000 GALLONS ADSORPTION TREATMENT**	CARBON REACTIVATION***	TOTAL
A*	128	Phenolics TOC	10 mg/l 250 mg/l	0.1 mg/l	0.85	0.80	1.65
B*	18	Phenolics TOC	40 mg/l 300 mg/l	0.1 mg/l	4.00	2.10	6.10
C*	183	Phenolics TOC	100 mg/l 600 mg/l	0.1 mg/l	0.35	0.80	1.15
D	11	TOC	900#/Day	60#/Day	3.50	3.45	6.95
E*	26	TOC	275 mg/l	Specific Toxicity Tests	4.35	2.45	6.80
F*	84	Color TOC	800 mg/l	Color Removal	1.50	0.25	1.75
G	55	Phenolics TOC	1700 mg/l 3000 mg/l	0.1 mg/l	1.00	2.00	3.00
H	55	TOC	1000 mg/l	Specific Toxicity Tests	2.00	9.00	11.00

* = Filtration Treatment also

** = Includes: Capital, Maintenance, Insurance & Utilities

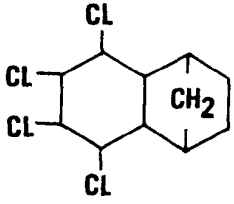
*** = Includes: Reactivation Capital, Transportation, Reactivation Utilities & Make-Up Carbon

MONS 064576

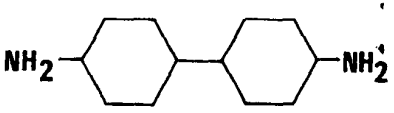
Figure 1

EPA PROPOSED LIST OF TOXIC ORGANIC CHEMICAL

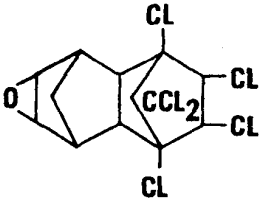
ALDRIN $C_{12}H_8CL_6$



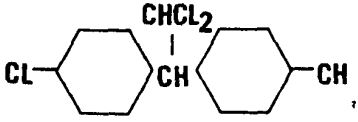
BENZIDINE $C_{12}H_2N_2$



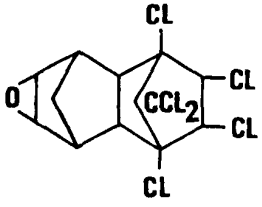
DIELDRIN $C_{12}H_8CL_6$



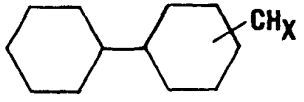
DDE (TDE) $C_{14}H_{10}CL_4$



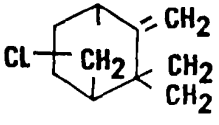
ENDRIN $C_{12}H_8CL_6O$



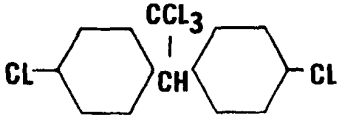
POLYCHLORINATED BIPHENYL
(210 Isomers)



TOXAPHENE $C_{10}H_{10}CL_8$



DDT $C_{14}H_9CL_5$



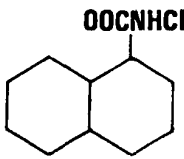
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Figure 2

TOXIC ORGANICS UNDER EPA EXAMINATION

(Partial List)

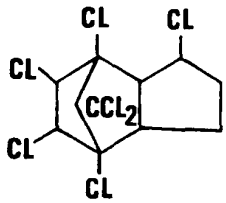
SEVIN $C_{12}H_{11}NO_2$



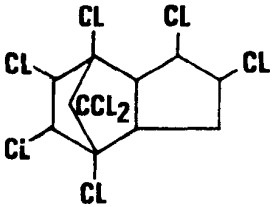
HYDROQUINONE $C_6H_6O_2$



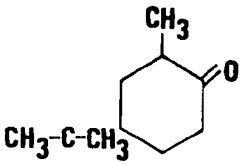
HEPTACHLOR $C_{12}H_5CL$



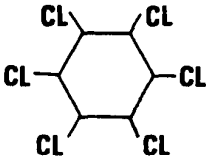
CHLORDANE $C_{10}H_6CL_8$



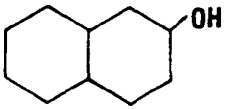
CAMPHOR $C_{10}H_{16}O$



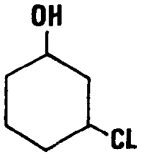
LINDANE $C_6H_6CL_6$



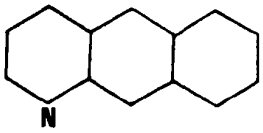
NAPTHOL $C_{12}H_8O$



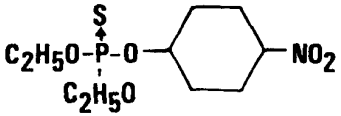
O-CHLOROPHENOL C_6H_5CLO



ACRIDINE $C_{13}H_6CL_6$



PARATHION $C_{10}H_{14}NO_5PS$



MONS 064578

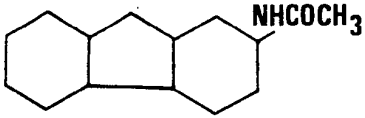
Figure 3

CARCINOGENS

(Partial List)

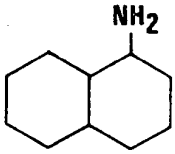
2-ACETYLAMINOFLOURENE

$C_5 H_{13} NO$



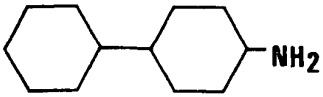
ALPHA-NAPTHYLAMINE

$C_{10} H_9 N$



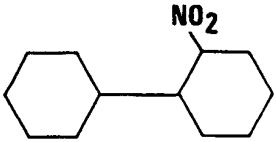
4-AMINODIPHENYL

$C_{12} H_{11} N$



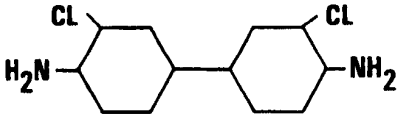
4-NITROBIPHENYL

$C_{12} H_9 NO_2$



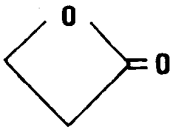
3,3'-DICHLOROBENZIDINE

$C_{12} H_{10} C_2 . 2 CHCL$



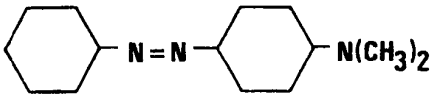
PROPIOLACTONE

$C_3 H_4 O_2$



4-DIMETHYLAMINOAZOBENZENE

$C_{14} H_{15} N_3$



ETHYLENIMINE

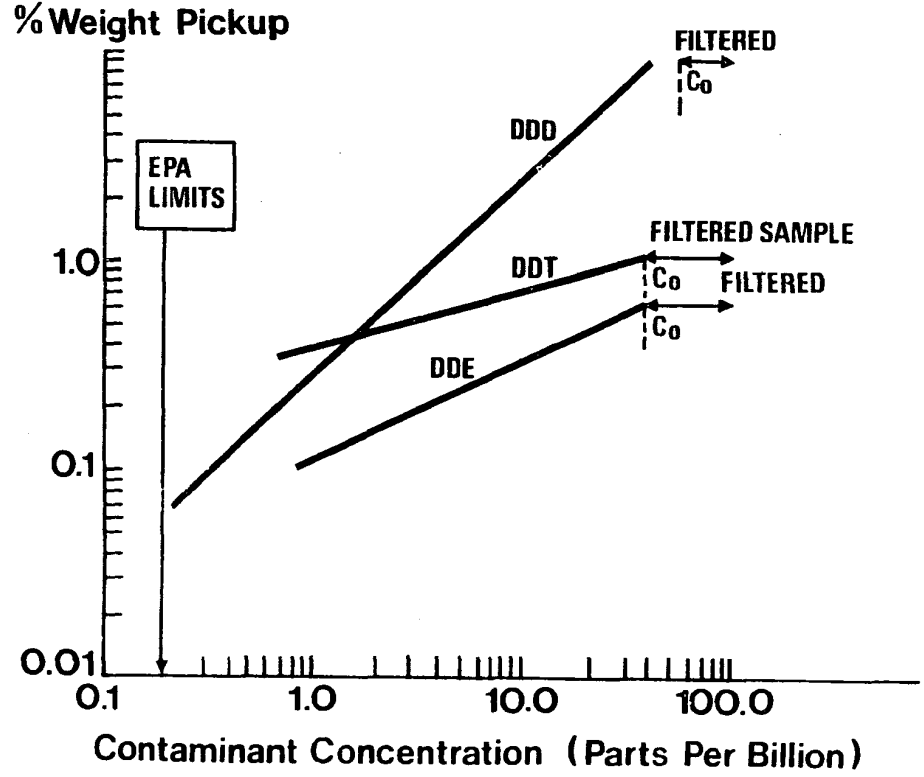
$C_2 H_5 N$



MONS 064579

Figure 4

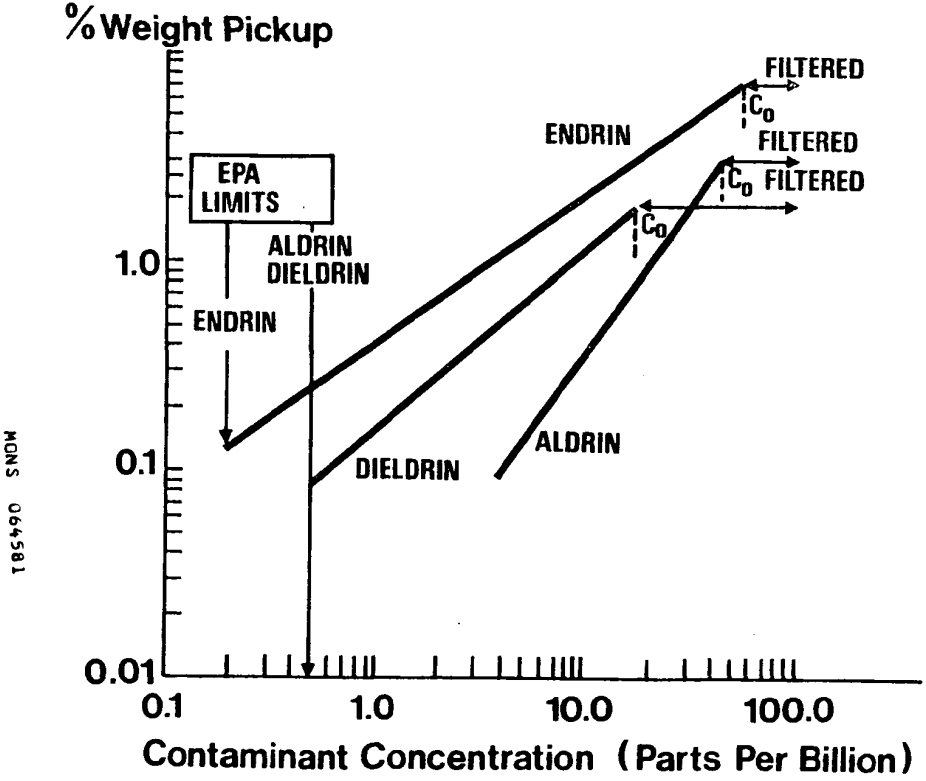
SIMPLIFIED ADSORPTION SUMMARY



MONS 064580

Figure 5

SIMPLIFIED ADSORPTION SUMMARY



MONS 064581

Figure 6

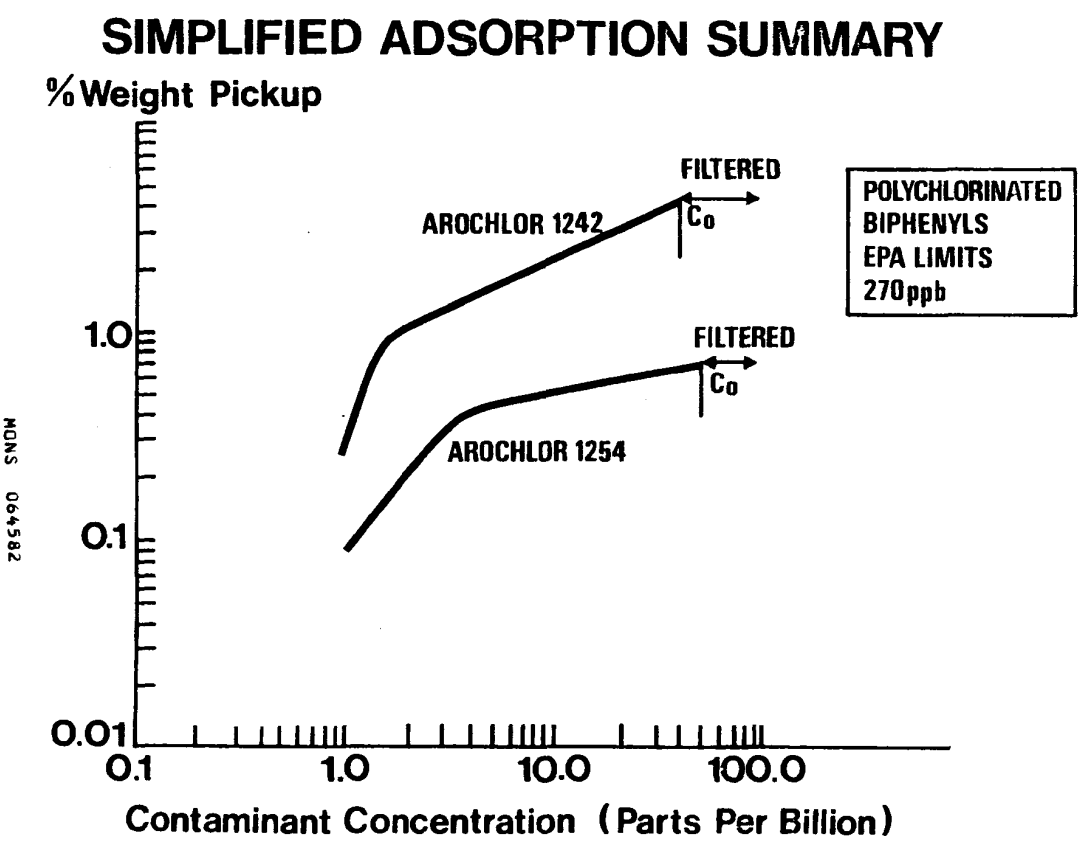
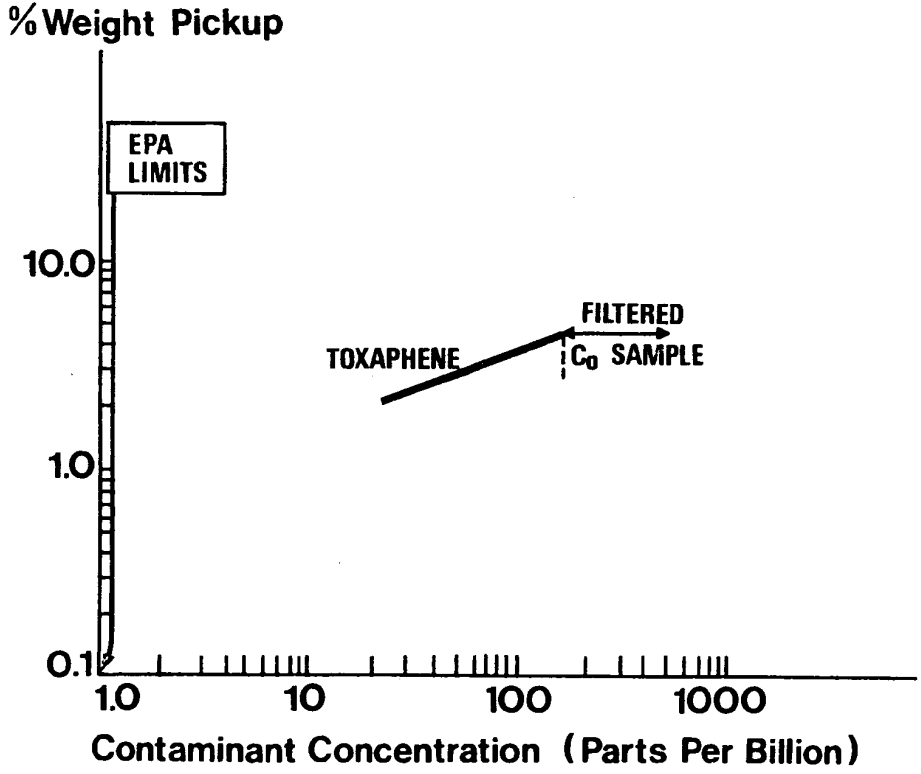

SIMPLIFIED ADSORPTION SUMMARY
% Weight Pickup
FILTERED
AROCHLOR 1242
Co
POLYCHLORINATED
BIPHENYLS
EPA LIMITS
270ppb
FILTERED
Co
AROCHLOR 1254
1.0
0.1
0.01
0.1
1.0
10.0
100.0
Contaminant Concentration (Parts Per Billion)
MONS 064582

Figure 7

SIMPLIFIED ADSORPTION SUMMARY



MONS 064583

Addendum 1

TOXIC CHEMICAL ISOTHERM AND ANALYSES PROCEDURE

Adsorption isotherms were run on aldrin, dieldrin, endrin, DDD, DDE, DDT, toxaphene and Arochlor 1242 and 1254. The first seven are pesticides, and the last two are mixtures of PCB's containing 42% and 54% chlorine by weight. Stock solutions of each compound, except toxaphene, were prepared in acetone at 100 mg/l. Toxaphene was made up at 1000 mg/l. Filtrasorb^{R} 300 (FS-300) activated carbon was used throughout. Carbon was added from a stock suspension of 1 g or 2 g of pulverized FS-300 per liter of distilled water.

Isotherms of aldrin, dieldrin, endrin, DDD and Arochlor 1242 and 1254 were prepared by the following method. Exactly 1 ml of stock pesticide or PCB solution was added to seven flasks, each containing slightly less than 1 liter of distilled water. Measured volumes of the 2 g/l carbon stock solution was added to each flask to give carbon concentrations of 0, 2, 5, 10, 25, 50 and 100 mg/l. The total volume of each flask was 1000 ml. After four hours agitation on a wrist shaker, each solution was filtered through 0.45 micro-millipore pads and stored in a refrigerator in quart amber glass bottles with Teflon-lined caps until analyzed.

The same isotherm procedure was followed with DDE, DDT and toxaphene, except the carbon stock as 1 g/l and carbon dosages were 1, 2.5, 5, 12.5, 25 and 50 mg/l. Also, only 0.5 ml of toxaphene stock solution was added to each flask.

A nickel 63-electron capture gas chromatograph was used for analysis. All samples were extracted and concentrated approximately 100 times before analysis. The method is described in the 1971 EPA report, "Methods for Organic Pesticide Analysis in Water and Waste Water."

MONS 064584

Addendum 2

ANALYTICAL LABORATORY RESULTS

CARBON DOSAGE (mg/l)	RESIDUAL (ppb)								
	ALDRIN	ENDRIN	DIELDRIN	DDT	DDD	DDE	TOXAPHENE	AROCHLOR 1242	AROCHLOR 1254
Control	48	62	19	41	56	38	155	45	49
1.0	--	--	--	41	--	34	147	--	--
2.0	26	15	6.3	--	6.9	--	80	7.3	37
2.5	--	--	--	21	--	29	--	--	--
5.0	15	3.4	2.4	3.7	3.7	12	31	1.6	17
10.0	12	1.5	1.1	--	2.2	--	2.7	1.1	4.2
12.5	--	--	--	<1	--	3.3	--	--	--
25.0	6.3	0.56	--	--	0.45	1.1	--	--	1.6
50.0	4.4	0.22	--	--	0.35	0.9	--	--	1.2

MONS 064585