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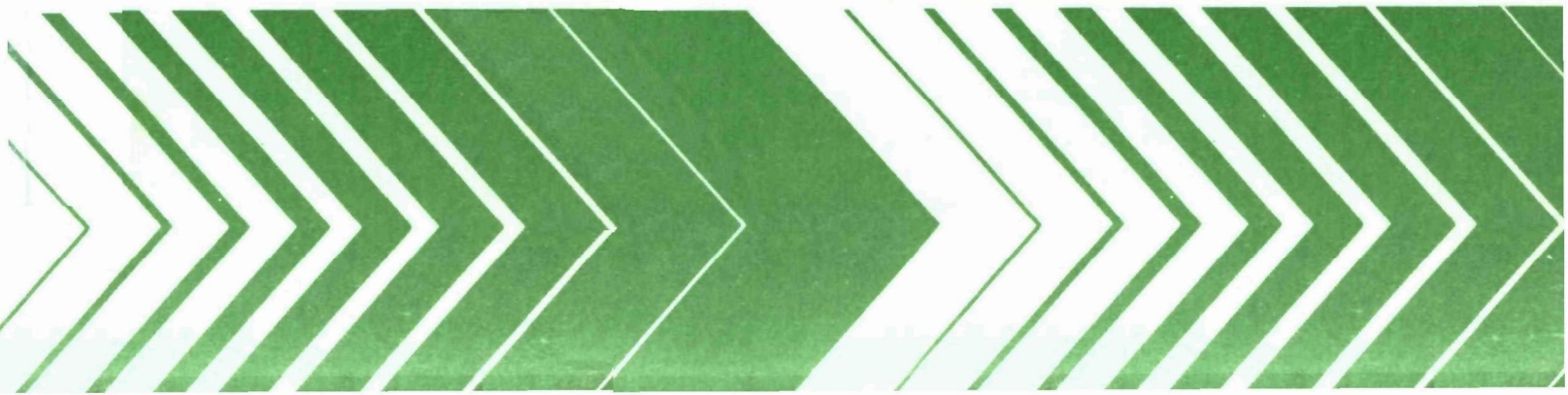
Application of Buoyant Mass Transfer Media to Hazardous Material Spills

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APPLICATION OF BUOYANT MASS TRANSFER MEDIA
TO HAZARDOUS MATERIAL SPILLS

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory-Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report describes a search for alternative means (other than the current ballasted-package approach) for dispersing mass transfer media in ponded water to clean up spills of hazardous materials. As a result of this search, a method to slurry buoyant activated carbon into static water bodies, such as lakes and reservoirs, was developed and successfully field-tested. In the process of development it was discovered that no reliable commercial source of buoyant activated carbon existed. Further research efforts resulted in development of a unique method of producing buoyant activated carbon. This report will be of interest to those responsible for cleanup of hazardous materials spilled in static water bodies, where direct application of mass transfer media is not effective because of dispersal problems. Information concerning this subject beyond that supplied here may be obtained by contacting the Oil & Hazardous Materials Spills Branch, IERL-Ci, U.S. EPA, Edison, New Jersey 08817.

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ABSTRACT

A prototype system was designed and developed to slurry buoyant acti-
vated carbon into a static body of water. The process was developed to
remove soluble hazardous compounds from a watercourse as the result of an
accidental spill or chronic problem.

The basic system was barge-mounted with an intake pump, a jet-slurrier,
a surge tank, and a slurry pump. The buoyant carbon was fed into the slurrier
by gravity from a floating, hopper-bottom tote bin.

In a simulated spill, up to 98% removal of Diazinon was achieved by
adsorption on activated carbon and by dispersion of the spilled material.

A unique method of making buoyant activated carbon was developed using
microballoons and a carbon coating mix to create small buoyant adsorptive
media. Estimated cost per pound of media was $3.50 on a small-batch basis.

No acceptable buoyant activated carbon is commercially produced in the
United States at this time, although the technology for production has been
developed.

This report was submitted in fulfillment of Contract No. 63-03-2204
under the sponsorship of the U.S. Environmental Protection Agency. The
report covers the period June 1975 to April 1977, and work was completed as
of September 1976.

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SECTION 1

INTRODUCTION

The use of buoyant activated carbon for the treatment of hazardous material spills in static and flowing water bodies has been reported previously (1-6). Addition to flowing water is accomplished by direct application to the surface of the stream. Natural turbulence in the water and subsequent turnover serve to stimulate intimate contact between the media and water. It is hypothesized that this contact results from two distinct mechanisms (4):

- Turbulent mixture of the media down into the water column

- Continual movement of more contaminated water to the surface as a function of turnover.

These mechanisms are largely absent from ponded waters such as lakes and reservoirs. Therefore, direct application to the surface of the water will have little effect in treating spills of soluble hazardous materials. Though the media would effectively cleanse the contaminated water near the surface, it has no means of concentrating contaminant from deeper in the water column. Consequently, the application of buoyant mass transfer media to ponded waters requires some means of stimulating contact between media and larger volumes of water if effective treatment is desired. In work reported to date (1-3), contact has been stimulated by gathering media in ballasted packages designed to release the media once the package rests on the bottom of the water body. Hence, contact occurs as the buoyant media rises in the water column.

Several difficulties have been noted with the ballasted-package approach. These include:

- The cost associated with packaging media in small ballasted units

- The residual packaging materials and ballast left in the water body after an application

- The difficulty in trying to obtain even coverage of affected waters with discrete packages.

The study reported here was designed to explore an alternate means of dispersing buoyant mass transfer media that would avoid or minimize the difficulties mentioned above. En route to that objective, it was

determined that no reliable source of buoyant activated carbon existed. Since activated carbon is the most universally applicable mass transfer agent, it was decided that development of the new dispersion system would be undertaken only if a supply of buoyant activated carbon could be assured. Consequently, the final objectives of the project were twofold:

- Identify commercially available buoyant activated carbons or a means of producing the same from available products

- Demonstrate the feasibility of applying buoyant activated carbon to ponded water with a barge-mounted slurry-pump device.

Laboratory and field investigations in pursuit of these objectives are detailed in the following document.

SECTION 2

CONCLUSIONS

Conclusions drawn from the study of buoyant activated carbon availability are as follows:

- Currently, there are no companies that manufacture a buoyant activated carbon although the technology to produce such media does exist.

- Carbon manufacturers would produce a buoyant activated carbon if a market was created for the product.

- There is an available method of producing floating activated carbon by bonding carbon and glass microballoons into small particles.

The removal of an organophosphorous pesticide, Diazinon, was effectively demonstrated in field tests using buoyant mass transfer media delivered into a water body through a slurry system. Other conclusions derived from the field test include:

- The slurry system is capable of delivering a wetted carbon slurry under water more effectively than aerial bombardment methods.

- Containerized bins can be used to ferry large volumes of buoyant carbon to slurry systems.

- The slurry system works better with slurrier and carbon feed above water, rather than below water.

SECTION 3

RECOMMENDATIONS

The slurry injection system tested in the subject project was found to be effective in dispersing buoyant media through a water body. As a result of this work, the following recommendations are offered:

System Application

The slurry delivery system should be considered the preferred technique for application of buoyant media.

Modifications of the prototype unit should be considered for use on dedicated barges in high incident areas. These units could perform emergency response as well as routine cleanup activities.

The slurry delivery system should be considered for use in fixed facilities downflow from high spill potential areas to provide on-line response capabilities.

System Design

Tote bins should be shorter, with ballast in the plenum zone, to facilitate upright partioning when floated.

Relief valves should be mounted on all bins to prevent pressurization.

The barge should be equipped with an A-frame device to allow vertical movement of bins onto the jet-slurrier such that operation is conducted above water.

Buoyant Media

Future development of buoyant media systems should be directed to identification and/or production of buoyant carbon until a source is assured.

Preference should be given to naturally buoyant particles that can be produced in existing facilities. While a composite media can be produced, it should not be pursued unless costs can be reduced by a factor of two or more.

SECTION 4

CARBON AVAILABILITY

BACKGROUND

    During the early development work with buoyant mass transfer media, it
was found that two commercially available carbons, Nuchar C-190 and
Nuchar WA, produced by Westvaco, were naturally buoyant. Both activated
carbons were produced from lignin materials found in the wastes from
Westvaco's pulp and paper production operations. Nuchar C-190 was selected
for subsequent field trials because it offered a greater fraction of buoyant
particles per batch. The approximate floatability of various mesh sizes is
detailed in Table 1. Further laboratory tests yielded the rise time charac-
teristics presented in Table 2 and the phenol adsorption isotherm given in
Figure 1.

TABLE 1. APPROXIMATE FLOATABILITY OF VARIOUS
          MESH SIZES OF NUCHAR C-190 (Approximate
          % Floating)

Mesh size	Hours of shaking time				
	1	2	4	6	24
20	100	100	95	95	85
20-30	95	95	95	90	85
30-50	95	95	95	90	85
50-100	95	90	90	90	85
100-200	90	90	85	80	80
200-325	80	80	75	70	70

    Since completion of the initial trials, the "feed stock" of Westvaco
carbons has been changed. Waste control measures at the paper plants have
eliminated the original lignin raw materials. While Westvaco has developed
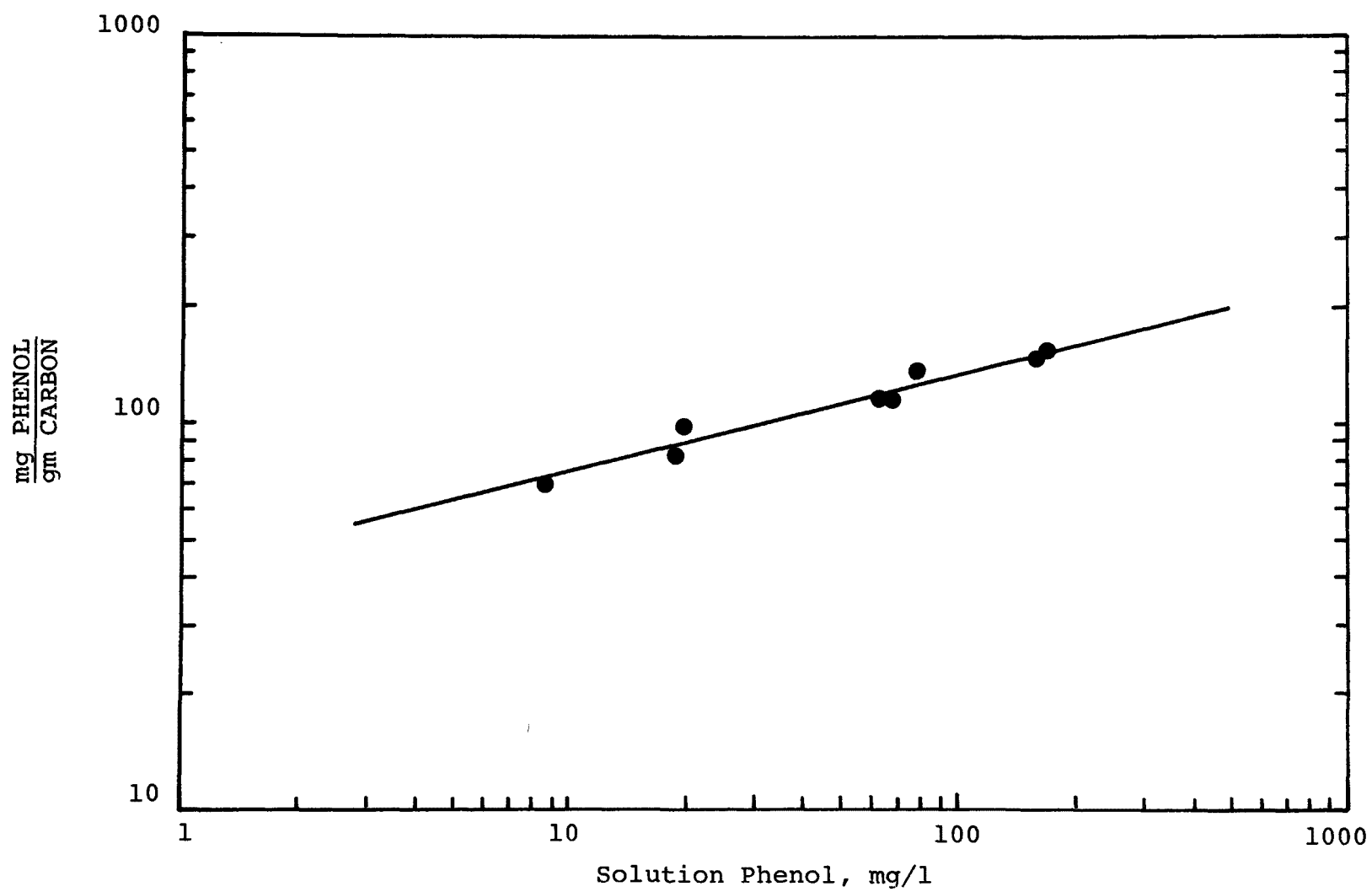
Figure 1. Sorption isotherm for phenol and Nuchar C-190.

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a line of replacement carbons, none have been found that maintain the buoyancy of C-190 and WA. Consequently, new sources of buoyant activated carbon were required if this technology was to be utilized. Pursuant to this, an investigation of possible replacements was initiated. Two alternatives were evident: 1) other buoyant carbons could be identified with capabilities comparable to C-190, or 2) means could be found by which nonfloating carbons could be rendered buoyant without sacrificing capacity.

TABLE 2. RISE TIME FOR NUCHAR C-190
IN 1.2m (4 ft) OF WATER

Mesh size	Time for 95% of carbon to surface (minutes)
30-50	5
50-100	10
100-200	15
200-300	15

COMMERCIALLY AVAILABLE BUOYANT CARBONS

Preliminary Screening

Contact with carbon manufacturers was first made with a letter of inquiry accompanied by research reports describing the use of buoyant media for treating hazardous material spills. This mailing was addressed to the 31 individual firms listed:

Aircon Corporation - 304 Carrousel Towers, Cincinnati, OH 45237

American Norit Company - 6301 Glidden Way, Jacksonville, FL 32208

AMF, Cuno Division - 400 Research Parkway, Meriden, CT 06450

Amoco Research Corporation, Standard Oil Company (Indiana) - 200 East Randolph Drive, P.O. Box 5910-A, Chicago, IL 60690

Barnaby-Cheney Company - 835 North Cassady Avenue, Columbus, OH 43216

Belco Pollution Control Corporation - 570 West Mt. Pleasant Avenue, Livingston, NJ 07039

Calgon Corporation - P.O. Box 1346, Pittsburgh, PA 15230

Catalytic Products International, Incorporated - 524 Mill Valley Road, Pallatine, IL 60067.

C-E Natco - P.O. Box 1710, Tulsa, OK 74101

Drew Chemical Corporation - 701 Jefferson Road, Parsippany, NJ 07054

EML Laboratories - 500 Executive Building, Elmsford, NJ 10523

Envirex, Incorporated - P.O. Box 6, Conshocken, PA 19428

Great Lakes Carbon - 299 Park Avenue, New York, NY 10017

Hampden Color and Chemical Company - 126 Memorial Avenue, Springfield, MA 01101

Harshaw Chemical Company - 1945 East 97th Street, Cleveland, OH 44106

ICI America Incorporated - Wilmington, DE 19899

Infilco Degremont, Incorporated - Martinsville Road, Liberty Corner, PA 07938

Ionac America Incorporated - Birmingham, NJ 08011

J. F. Henry Chemical Company - 245 Park Avenue, East Rutherford, NJ 10020

J. T. Baker Chemical Company - 222 Red School Lane, Phillipsburg, NJ 08865

Kisco Boiler and Engineering Company - P.O. Box 328, 1917 Rutger Street, St. Louis, MO 63104

L. A. Salomon and Br. Incorporated - P.O. Box 828, Port Washington, NJ 11050

Nuclear Supply Company - 422 Washington Building, 15th and New York Avenue, N.W., Washington, DC 20005

Phillips Manufacturing Company - 7334 Clark Street, Chicago, IL 60626

R. W. Greff and Company, Incorporated - One Rockefeller Plaza, New York, NY 10020

Shawnigan Products Corporation - 111 Charlotte Place, Englewood Cliffs, NJ 07632

Techni-Chemical, Incorporated - 205 East State Street, P.O Box 428, Cherry Valley, IL 61016

Union Carbide Corporation - Carbon Production Division, Material Systems Division, 270 Park Avenue, New York, NY 10017

Westvaco Corporation - Chemical Division, Covington, VA 24426

Witco Chemical Company - 277 Park Avenue, New York, NY 10017

Seven firms responded positively and submitted samples for testing. In addition, Barnaby-Cheney and Union Carbide indicated that they produce pelletized carbon products which could potentially have buoyancy agents added to effect floatation. It was later determined that these pellets were too large to be of use and both producers were reluctant to modify production facilities to meet programmatic needs. Finally, Union Carbide indicated that they produce a carbon coating mix of powdered activated carbon and adhesive which could be bonded to various substances.

Qualitative floatation tests were conducted on all samples received to determine those that warranted further quantitative evaluations. Results of this initial screening work are presented in Table 3. Additional testing of the Witco CKD revealed that it would not wet and therefore would have little capacity in aqueous systems. Therefore, attention was focused on the floating samples provided by ICI, Amoco, and Union Carbide.

TABLE 3. QUALITATIVE FLOATABILITY SURVEY OF CARBON SAMPLES

Company	Sample	Results
Barnaby-Cheney	NW9468	No Floatation
	UU9588	No Floatation
	NL	No Floatation
Westvaco	Nuchar S-A	No Floatation
ICI America	S51 + Kerosene	Good Floatation
	S51 + No. 2 Fuel Oil	Good Floatation
Amoco	Original	Good Floatation (85%)
	C-114	No Floatation
American Norit	FQA	No Floatation
	A	No Floatation
	EX	No Floatation
	F	No Floatation
Union Carbide	High surface area	No Floatation
	Low surface area	Good Floatation
Witco	CKD	Floats, but hard to wet
	CLD	5% Floaters
	ACD	10-20% Floaters

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Laboratory Testing

In order to provide comparative data on prospective carbons with respect to the performance of Nuchar C-190 employed in the original studies, a series of quantitative floatability and rise time evaluations were conducted. Test methodology was identical to that reported for the Nuchar C-190 (2). For floatability analysis, samples were divided into small lots according to mesh size. Each sizing was then slurried in water and shaken for a preset time interval after which mixtures were allowed to stand for five minutes. At that point, visual observations were made of the amount of carbon that had risen to the surface. Results of this analysis on test carbons are presented in Tables 4 through 6. These data can be compared directly with those for Nuchar C-190 given in Table 1. The Union Carbide LSA carbon is <325 mesh but offers almost 100% floatation. All carbons tested are acceptable from a floatability standpoint, but the LSA and Amoco are superior to the treated S-51 carbons.

TABLE 4. FLOATATION CHARACTERISTICS OF CANDIDATE
CARBON: V-AMOCO (% Floating)

Mesh size	Hours of shaking time				
	1	2	4	6	24
Whole C	85	85	80	NA	NA
50-100	85	85	80	80	80
100-200	80	80	70	65	65
200-325	85	85	80	80	80
<325	85	85	85	85	85

TABLE 5. FLOATATION CHARACTERISTICS OF CANDIDATE
CARBON: VI - ICI S51 AND KEROSENE
(% Floating)

Mesh size	Hours of shaking time				
	1	2	4	6	24
Whole C	70	50	50	50	50
50-100	70	70	65	65	50
100-200	75	70	65	65	50
200-325	50	50	50	50	50

TABLE 6.   FLOATATION CHARACTERISTICS OF CANDIDATE
CARBON: VII - ICI S⁵1 AND NO. 2 FUEL OIL

	Hours of shaking time				
Mesh size	1	2	4	6	24
Whole C	70	50	50	50	50
100-200	70	70	70	60	50
200-325	50	50	50	50	50
<325	75	70	70	70	50

The relationship between mesh size and rise time was investigated using a glass column measuring 1.3 m in length and 12.7 cm in diameter. Samples of carbon in various mesh sizes were placed on the water surface and the column inverted. The time required for the bulk (>95%) of the carbon to rise through the water column to the surface was recorded. Results of these experiments are presented in Tables 7 through 9 and can be compared directly to the data on Nuchar C-190 in Table 1. Once again, the size uniformity of Union Carbon LSA precluded the testing of various size ranges. The fine powder was found to reach the top of the rise time column in an average of 3 to 5 minutes. The Amoco carbon displayed very favorable rise time characteristics. Treated S-51 carbons rose very quickly.

TABLE 7.   AMOCO

	First large concentration to top	Carbon evenly distributed in column	50% to top	90% to top
Mesh size				
Whole C	4	7	12	25
50-100	2	4	7	18
100-200	3	6	12	27
200-325	6	7	13	30
<325	5	6	11	27

Comparison of capacities was achieved through the use of standard adsorption isotherms derived from batch contact experiments. The procedure involved mixing various measured weights of carbon with a standard solution of 1000 mg/l methyl isobutyl ketone (hexone) which yielded a baseline of 1850 mg/l TOC. Contact was maintained for a 24-hour period to assure that equilibrium was reached. At that time, samples were filtered and analyzed for total organic carbon content. A blank containing no carbon was employed to allow measurement of total hexone adsorbed in each case. Results of these runs are compared to the isotherm for Nuchar C-190 in Figure 2.

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TABLE 8. ICI S51 AND KEROSENE

Mesh size	50% to top	90% to top
Whole C	.75	1.5
50-100	.5	1.3
100-200	.75	1.7
200-325	.75	1.7

TABLE 9. ICI S51 AND NO. 2 FUEL OIL

Mesh size	50% to top	90% to top
Whole C	1	2.5
100-200	.83	2
200-325	1.2	2.5
<325	1.5	2.6

Results and Discussion

From the isotherm data, it is clear that the S-51 samples have virtually no capacity. The use of kerosene and No. 2 fuel oil for buoyancy has saturated the adsorption sites. Therefore, these carbons are of little value for spill treatment. The Amoco and LSA samples, however, compare very favorably with Nuchar C-190. In light of these properties, it was determined that either the Amoco or the LSA carbon would be acceptable as a replacement for the original Nuchar C-190.

After selection of the two carbons, Amoco and Union Carbide personnel were contacted to determine the production status of the carbons. At that time, Amoco announced that they had switched their production techniques to generate a heavier, more easily wetted carbon. When the latter was tested, it was found to display none of the required buoyancy properties. While Amoco still has the technology to produce the buoyant carbon, they have chosen not to pursue it until a substantial market develops. The buoyant carbon is undesirable for the market Amoco is presently addressing.

The Union Carbide LSA carbon is produced from an industrial waste stream. The process facility that generates this waste will be on-line within the next year. Should there be a market for the buoyant carbon at that time, Union Carbide can perform the appropriate treatment sequence to produce the LSA. Production can be accomplished with existing equipment.

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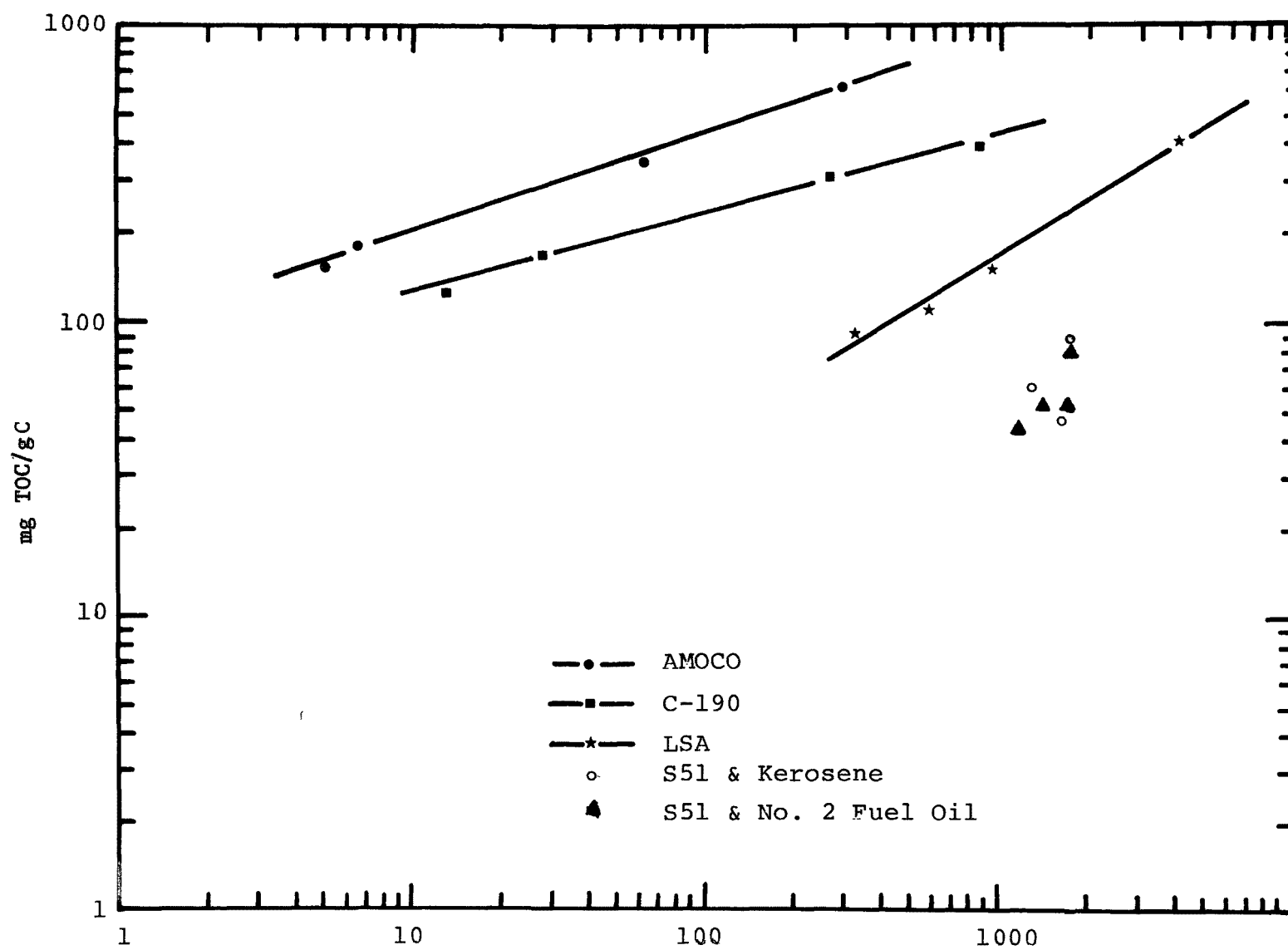
Figure 2. Milligrams TOC remaining in solution.

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Since the facility is not presently operating, however, it was not possible to obtain large samples of LSA for the field demonstration of the slurry discharge system. Therefore, alternate sources of buoyant carbon were still required.

BUOYANT CARBON COMPOSITES

Preliminary Studies

At this point, the strategy for making buoyant carbons was changed to one of seeking means of combining available carbons with naturally buoyant agents such that the composite would float, but retain the carbon's capacity. This approach requires the presence of a binding agent capable of holding the aggregate particles without eliminating a significant portion of the active surface. Early efforts centered on the use of materials that could serve the dual role of binder and floatation agent. Major candidates were the low density plastics such as polypropylene, polyethylene, and polystyrene. Small granules of these plastics were mixed with a powdered activated carbon and heated to the pour point. At that time the tacky mixture was quickly cooled and buoyant particles collected through floatation. In all cases, low density carbon-coated granules resulted. The product, however, was unacceptable. The polyethylene and polypropylene-based samples proved to be too hydrophobic. They resisted wetting, and therefore performed poorly in adsorption tests. The polystyrene-based samples were brittle and easily broke apart with agitation. In addition, the carbon fraction of the composite was generally small.

Carbon Bound To Microspheres Concept

At this point, attention was redirected to composites utilizing a separate binder. As noted earlier, Union Carbide produces a proprietary carbon coating mix which contains an inert binder capable of attaching the fine carbon particles to virtually any substrate. Samples were obtained, dried,

TABLE 10. HEXONE REMOVAL EFFICIENCY OF
DRIED CARBON COATING MIX

Grams of carbon	Carbon/hexone ratio	% Removal
Control	–	–
1.25	–	61
2.5	–	92
5.0	–	98
7.5	–	98.5

and ground into granules for testing. Results of a 24-hour shaker test with 200-ml solution columns containing 1480 mg/1 hexone are given in Table 10.

The dried coating mix showed excellent capacity for hexone removal. Apparently, the binder does not interfere with active sites to any significant extent. Next, batches were produced with hollow glass microspheres to see if the adhesive would form a strong enough bond to survive abrasion in a slurry pump. While buoyant granules were easily produced at low drying temperatures, immersion in water weakened the binder causing the carbon to separate and sink. Production at higher temperatures resolved this, but led to such hard particles that the microspheres were crushed when product was ground to the appropriate mesh sizes. These difficulties were circumvented with the use of the following four-step process:

1. Sufficient volumes of microspheres and carbon coating mix were uniformly mixed.

2. This mixture was oven dried at 105 to 110°C. (This hardens the binder but does not cure it.)

3. The product cake was crushed to the desired particle size range. (Relatively light crushing pressure can be used on the uncured particles and hence, the integrity of the microspheres can be maintained.)

4. The particles were cured at 180°C for 1 hour. (This set the binder to a state that was highly resistent to abrasion and water. Higher temperatures destroy the binder.)

Production with the staged process yielded particles with a high fraction of buoyant particles, good integrity, and excellent sorption properties.

Given the above observations, it was determined that the carbon coating mix-microsphere composite was an acceptable substitute for buoyant carbon, which could be available for field demonstration. Therefore, preparations were made to produce 454 kg (1000 lb) of composite particles. Four types of commercially available microspheres (characterized in Table 11) were evaluated for use in the composite. The lowest cost microspheres with excellent resistance to shear stress, B 38/4000, were selected for use. Based on the pertinent densities, 49.9 kg (110 lb) of microspheres are required to produce 454 kg (1000 lb) of carbon particles.

Subsequently, the 454-kg (1000-lb) batch of composite carbon particles was produced. The carbon coating mix was diluted slightly with water to enhance its fluidity. Microspheres were then added and blended in thoroughly with a small electric mixer. No large drying devices were available to dewater the resulting slurry, so the blend was spread on plastic sheets at a 1.27-cm (0.5-in.) thickness and dried with space heaters. The air-dried sheets of carbon were then placed in vibrating screens (10 mesh) and hand crushed. The particles were ground by the screen to a much finer size than 10 mesh, however. This, in part, was a result of the use of air drying, which left the binder weaker than desired. Optimally, a drier capable of 1.5 to 110°C should be employed. Curing was accomplished by placing the product in trays and heating it in muffle furnaces overnight.

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TABLE 11. CHARACTERISTICS OF COMMERCIALLY AVAILABLE MICROSPHERES

Sample	Floatation	Strength	Density	Cost/lb (>100 lb order)
Eccospheres FTD 202 (Emerson & Cumings, Inc.)	Excellent	Excellent	0.28 g/cc	$10.95
Q Cel pF (Philadelphia Quartz)	Some sinkers after 24 hours	Fair	0.19 g/cc	1.23
B 38/4000 (3M Corporation)	Excellent	Excellent	0.39 g/cc	3.55
Phenolic (Union Carbide)	Excellent	Fair	0.20 g/cc	2.15

Strengh test consisted of 5-min contact of water slurry in a blender followed by observation of fraction restoring floatation ability.

A qualitative evaluation of the final product revealed about a 50% floater fraction. Microscopic observation revealed that this resulted from a large amount of detachment between the microspheres and the carbon. This appears to have resulted from the low drying temperature and the grinding action of the screens. The binder was not sufficiently set to withstand the shear stresses generated during the crushing operation.

An estimated cost of buoyant media is approximately $7.64/kg ($3.47/lb) based only on costs of raw materials. A detailed cost breakdown is shown in Table 12. The price may be reduced if materials are purchased in bulk.

TABLE 12. FLOATING CARBON COST DATA

Raw Materials	Price/Unit	Total lb	Total
3M microspheres			
B38/4000	$335/cwt	200	$670
Carbon coating		1400	
mix 50% carbon	1.75/lb	700	2450
		carbon	
Total floating			
carbon made	3.47/lb	900	3120

SECTION 5

THE SLURRY INJECTION SYSTEM

PROTOTYPE DESIGN RATIONALE

The optimum approach for response to a spill provides for the rapid deployment and maneuverability of equipment. It was envisioned that a slurry system be constructed on a barge with all phases of media application included to achieve this optimum. At the same time it had to be kept in mind that of primary importance when handling the carbon, is the method of wetting the carbon while minimizing the amount of dust produced.

Carbon Wetting

There were several design options to consider to ensure that the carbon would be sufficiently wetted prior to introduction into the watercourse. Among these options were:

- A container of carbon that could be flooded with water, then pumped to deliver the resultant slurry

- A large bulk storage bin with pnuematic transport of carbon to a mixing vessel

- A bagged or drummed carbon fed through a hopper to a mixing vessel

- A self-contained feeder-slurrier system which could both feed and slurry the carbon in one operation.

The first option appeared to offer the simplest procedure. The concept relies on a watertight container valved to enable filling the container with water, while releasing air pressure resulting from the filling. Once the container is full the carbon and water can be mixed to completely wet the carbon. This slurry can then be pumped into the watercourse to effect organic removal. Unfortunately, difficulties are inherent in this process. While filling the container, the carbon wetted at the water-carbon interface forms a bridge in the container. When more water is added, there is a large increase in pressure, finally resulting in a blowout. The bridging problem can be eliminated by the addition of a mixer in the container, but there are questions of practicality in large-scale response systems.

The next option considered was that of utilizing a large bulk carbon bin with pnuematic transport of carbon from the bin into a water-carbon contactor from which the resultant slurry is pumped into the watercourse.

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This option is very attractive for a large-scale fixed-facility cleanup system. During spill response, however, there are too many time delays in transportation, setup, and operating procedures for this concept to be operational. There are also potential major problems with the system from carbon dusting associated with pnuematic transport. For these reasons this type of system was not considered further.

The least complex option, but that requiring the most manpower, is the simple addition of drummed or bagged carbon into an open mixing vessel followed by the pumping of the carbon slurry into the water. This approach also suffers from dusting problems during carbon addition. The procedure is virtually impossible to perform without producing clouds of suspended carbon. Another problem is the potential unavailability at the site of the large amount of manpower required to transfer large quantities of carbon in small containers.

The final option was deemed most favorable. It involves the incorporation of a feeder slurrier which, in operation, would eliminate dusting, manpower problems, and also require the least amount of maintenance. A Halliburton Jet-slurrier was chosen for this function. Jet-slurriers were originally designed and developed by Halliburton to rapidly wet and mix cement for sealing and setting oilfield casings. The Jet-slurrier is operated by forcing high pressure (7000-14,000 g/sq cm (100-200 psi) water through a jet into a small open mixing chamber, and out of the slurrier. The pressure differential across the opening creates a vacuum which is utilized to feed and mix dry solids into the water. The negative pressure tends to eliminate dusting problems inherent in fine dry solids mixing. The jet in the slurrier can be adjusted to different size openings for different influent pressures and performance (Figure 3).

Carbon Transport and Introduction

With the selecton of the jet-slurrier as the central part of the slurry system, the design effort turned to identification of the method of introducing the carbon into the slurrier. The simplest method of feeding the slurrier is gravity flow into the mixing bowl. A large mobile container was sought that would meet the criteria for the system. A hopper-bottom tote bin manufactured by Tote Systems Incorporated met all criteria. This bin (an A-90 hopper-bottom tote bin) has a capacity of 2040 ℓ (72 ft^3), a built in hopper, and a watertight butyl rubber butterfly valve which can be operated remotely (Figure 4). The bins are made of aluminum and are certified as watertight. Field trials have shown that the bins can be floated in water while full of buoyant carbon.

While the bins do float in water, they were very unstable when floating upright. This necessitated drilling holes into the airspace between the hopper and the bin wall, and flooding with water. The tote bins then became more stable and floated upright. Rubber stoppers were used to plug the holes to prevent water from flowing out of the flooded space when the tote bins shifted while floating. Even with the flooded area, the bins did not float well. Future design work should concentrate on weighting the bottom of the bins and/or making the bins shorter. This would make a much more stable bin and facilitate positioning the bin over the slurrier.
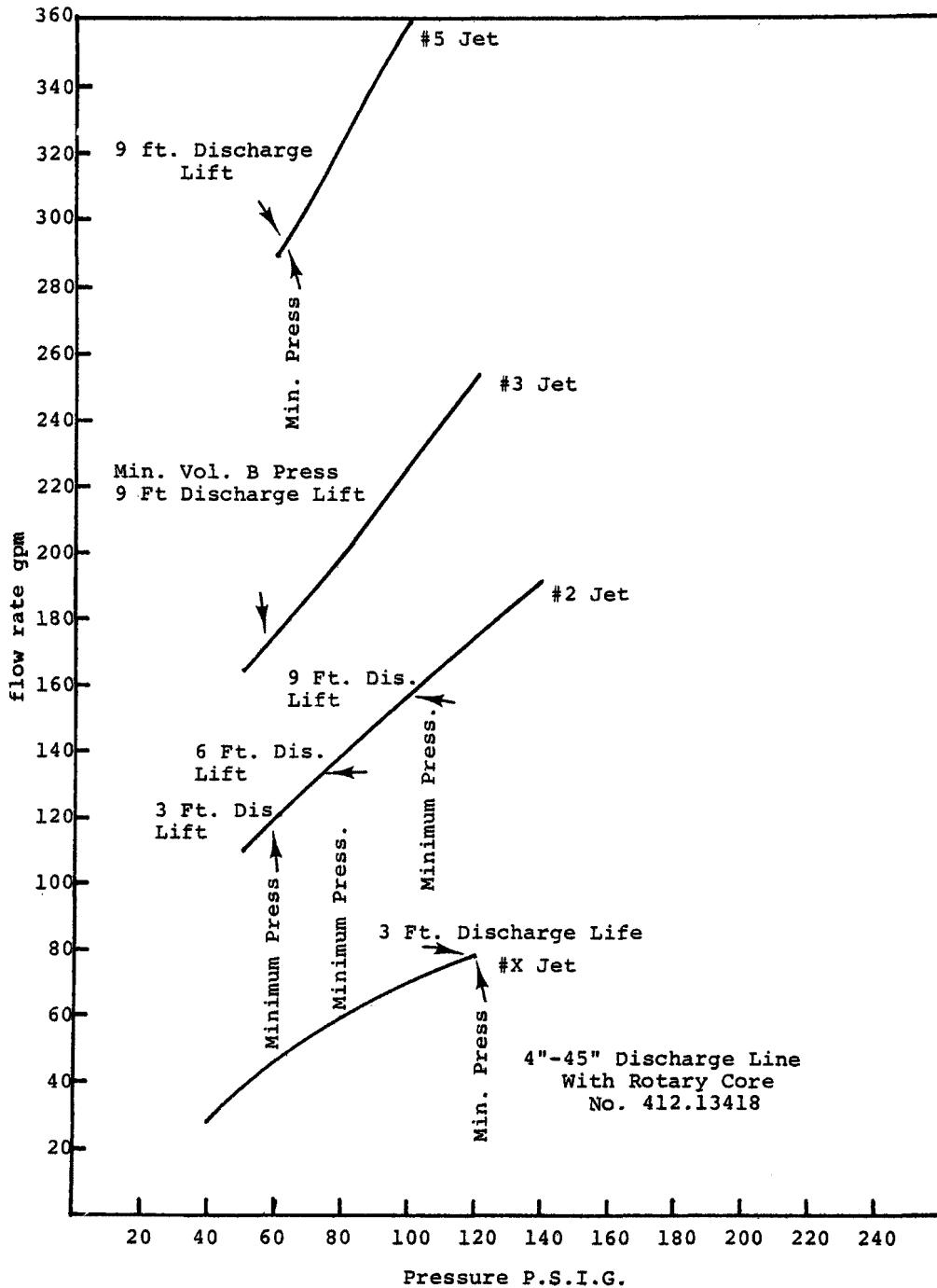
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Figure 3. Jet-slurrier performance curves.
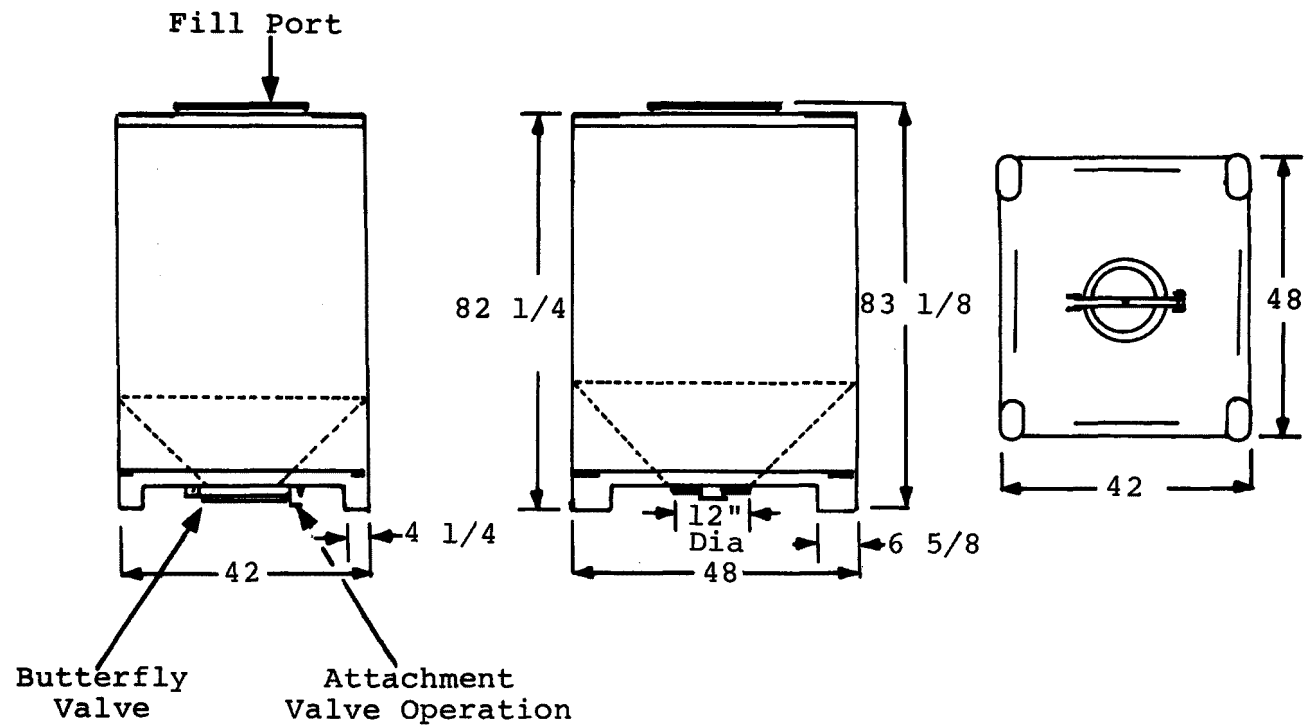
Figure 4. Layout and dimensions of tote bin. (dimensions in inches)

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The use of floating bins was incorporated into the prototype system as a means of transporting large amounts of the carbon without having to carry the entire inventory on a large, cumbersome barge. In an operational system, one can envision a train of full tote bins pulled along behind the barge, and successively placed upon the slurrier, exhausted, and removed. In a large spill, empty bins could be ferried to shore, filled with carbon, and placed back into the train thus allowing continuous operation of the slurry system.

System Integration

In order to integrate the two main components, it was necessary to modify the Jet-slurrier and tote bins after receiving them from the manufacturers. A female receptacle was welded to the carbon inlet on the slurrier. The latter was designed to receive a 15-cm (6-in.) diameter male pipe attached to the outlet on the tote bin. A rubber gasket was attached to the slurrier to give a watertight seal when the tote bin was connected to the slurrier.

Two pumps are necessary to run the system. One pump must deliver water at pressures up to 14000 g/sq cm (200 psi) to run the slurrier and the other pump must be capable of pumping the carbon-slurry from the surge tank which receives the slurrier output. The two pumps selected for the prototype were a Marlow 6.3-cm (2 1/2-in.) fire pump for high pressure feed water and a 7.6-cm (3-in.) Hydromatic self-priming trash pump.

Since the Jet-slurrier, at optimum performance, can deliver only a 3-m (9-ft) head, a surge tank between the slurrier and the slurry pump was used. The barge was designed to have the 758-ℓ (200-gal) surge tank (3 m x 3 m x 3 m) below water when full. This eliminated added weight to the barge load, and it also allowed operating the slurrier at different throat sizes.

Flexible rubber hoses rated to the working pressures of the pumps were acquired to plumb the system. Camlock quick connect-disconnect fittings were banded onto the hoses to speed up system deployment.

PROTOTYPE DESCRIPTION

A flow chart of the injection system is depicted in Figure 5. Water is drawn into a high pressure fire pump and pumped into the Jet-slurrier at pressures up to 14000 g/sq cm (200 psi); the pressure differential pulls carbon into the slurrier and intimately mixes the carbon and water to form a wetted carbon slurry; the slurry leaves the slurrier and enters a surge tank for flow equalization; a slurry pump draws from the surge tank and pumps the slurry into the water column.

Floatation Barge

A make-shift barge on which to mount the system was constructed as depicted in Figure 6. The strength members were 5 x 15-cm (2 x 6-ft) fir covered with 1.3-cm (0.5-in.) exterior plywood decking. Transportation

Carbon

```
              ┌──────────┐
              │   Tote   │
              │   Bin    │
              └──────────┘
                    │
                    ▼
┌──────────┐   ┌──────────┐        ┌──────────┐        ┌──────────┐
│   High   │   │   Jet-   │ Slurry │  Surge   │        │  Slurry  │  Into
Intake ──▶│ Pressure │──▶│ Slurrier │───────▶│   Tank   │──────▶│   Pump   │──▶ Water
│   Pump   │   │          │        │          │        │          │  Course
└──────────┘   └──────────┘        └──────────┘        └──────────┘
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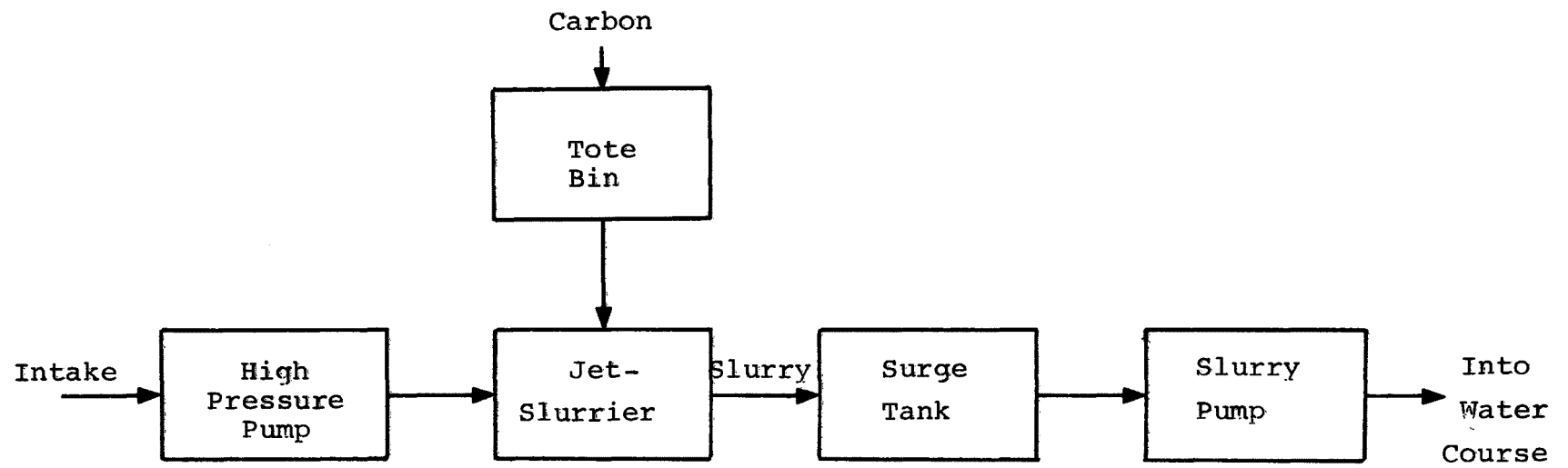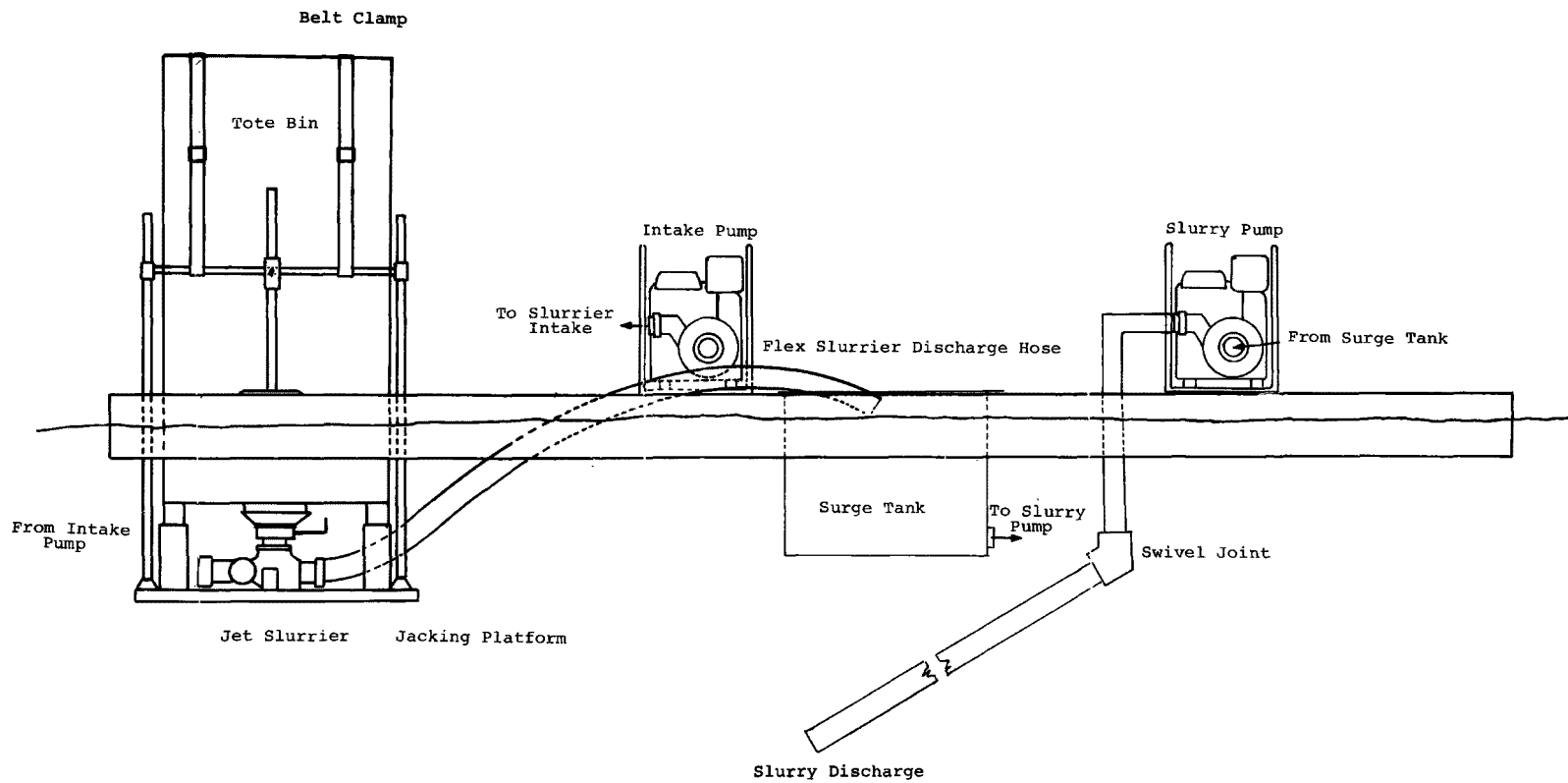
Figure 5.   Flow chart of slurry system.

Figure 6.   Layout of slurry delivery barge.

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constraints required that no piece of the barge be more than 2.4 m (8 ft) wide. This necessitated a two section configuration that was inherently weak. One section was 2.4 x 5.2 m (8 x 17 ft) with the two pumps, surge tank, and jacking platform mounted on the section. Floatation drums, as described below, were mounted to the platform wherever possible to keep the section afloat. The second section was 1.2 x 5.2 m (4 x 17 ft). Sections were bolted together with 1.3 x 17.8-cm (1/2- x 7-in.) bolts, resulting in a weak point along the connection.

The floatation drums that were attached to the bottom of the barge were Dayton floatation drums manufactured by Dayton Marine Products. Each drum had a rated floatation of 136 kg (300 lb). Sixteen drums were attached to the barge to support a total weight of 2179 kg (4800 lb).

Jacking Platform

The final jacking platform was made of aluminum plate and aluminum angle bar as shown in Figure 7. The platform was raised and lowered by automobile bumper-jacks attached to the barge. When the jacking platform is raised, the 7.5-cm (6-in.) male tube on the bottom of the tote bin enters the slurrier opening and is sealed with a rubber gasket. The slurrier and platform in the raised position affect a watertight seal, which makes it possible to slurry under water.

HYDRAULIC TESTING OF PROTOTYPE

Tests were conducted in a 38,000-m^3 (10,000,000-gal) standby sedimentation basin maintained on the Hanford Atomic Energy Reservation. Field trials were designed to verify that the slurry system and barge would operate in simulated spill conditions. During pilot runs, it was possible to slurry water through the Jet-slurrier while the slurrier was submerged and then into the surge tank. The slurry pump worked well although it had a greater capacity than the fire pump. By adjusting the throttle on both pumps it was possible to maintain a constant flow through the system.

The slurrier was then tested to determine its ability to slurry carbon above water while the tote bin was suspended over it. A bin of carbon was positioned over the jacking platform and the latter unit raised until the weight pressure of the bin formed a tight seal on the receiving cone for the slurrier. The valve was then opened to allow entry of the carbon into the intake water stream. A wetted slurry resulted which was easily delivered into the basin. Two design features of the jacking platform are worthy of note. By accomodating underwater fuel with the slurrier, the platform is not required to bear the full weight of the bin. Rather, it bears only sufficient weight to create a seal and allows natural buoyancy to bear the balance. Secondly, straps are necessary to hold the bin in place so that as the carbon is exhausted, the bin's added buoyancy does not break the seal.
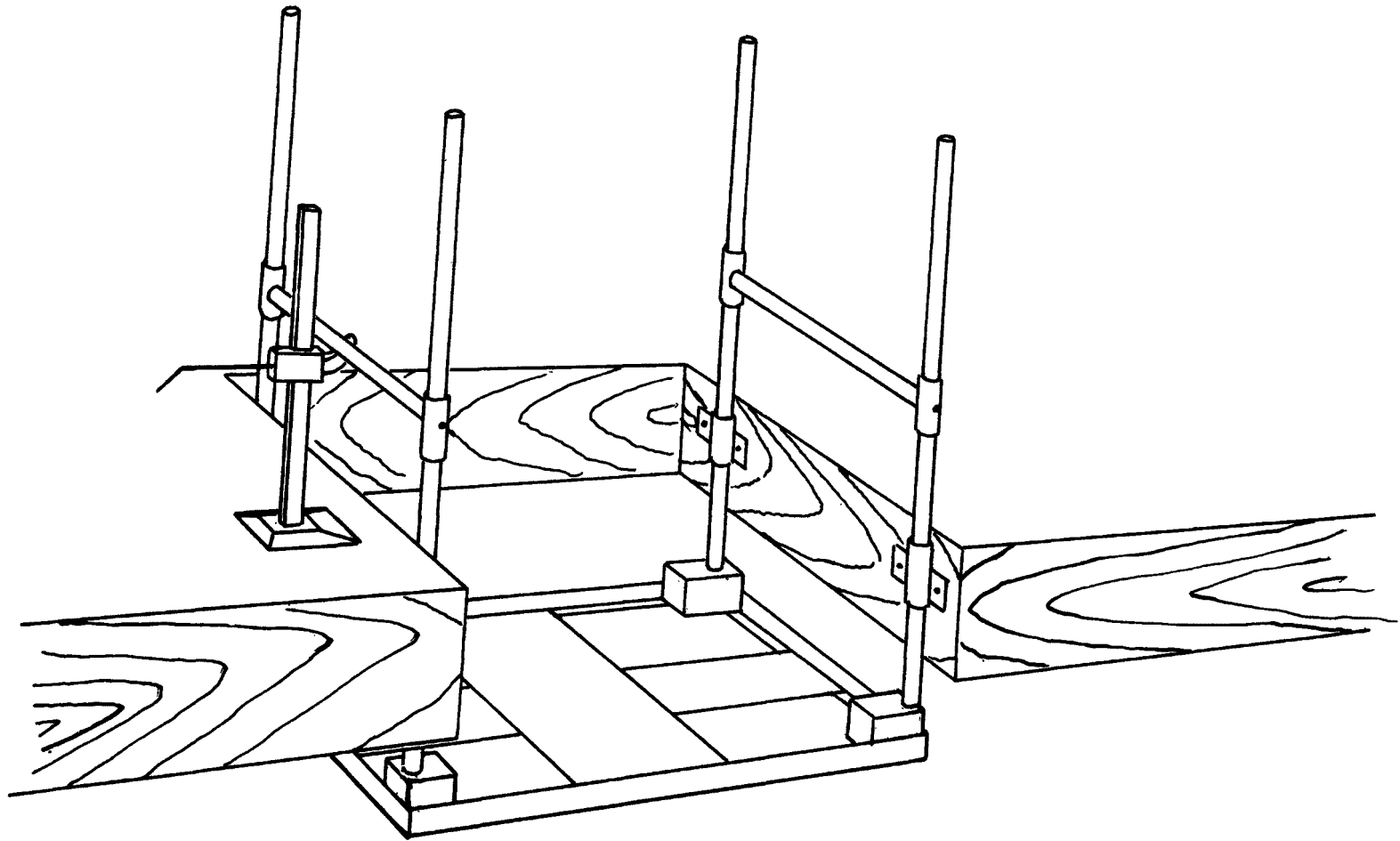
Figure 7. Layout of jacking platform.

SECTION 6

FIELD DEMONSTRATION

TEST PLAN

In order to test the effectiveness of the slurry dispersion system for application of buoyant media under the conditions of actual hazardous materials spills, field demonstrations were conducted. Conditions were selected to parallel a similar demonstration conducted with aerial application techniques as described by Mercer et al. (2). In this way, results would be directly comparable to those for the other application mode and objective selections could be made between the two.

The 38,000-m^3 (10-million gal) water storage basin employed previously was selected for the spill treatment demonstration. The basin was filled with Columbia River water to a depth of 3 m (10 ft) and calibrated ropes were placed across the western end of the 61- x 122-m (200- x 400-ft) structure as illustrated in Figure 8.

Once again a commercial grade of the organophosphorus pesticide, Diazinon, was selected for the spill. Diazinon is highly toxic to aquatic life forms and hence can cause major concern when spilled (2). It is not persistent beyond several weeks, however, so immediate, complete cleanup by the buoyant carbon was not required in the event discharge of the basin water was required at some future date (1). The composition of the Diazinon emulsifiable concentrate that was employed during each test is given in Table 13.

TABLE 13. COMPOSITION OF EMULSIFIABLE DIAZINON SOLUTION

Ingredient	1976 Test (%)	During Buoyant Carbon Test 1972 Test (2) (%)
0,0-Diethyl 0-(2-isopropyl-4-methyl-6-pyrimidyl phosphorothioate	49	48
Xylene	39	36
Inert ingredients	12	16

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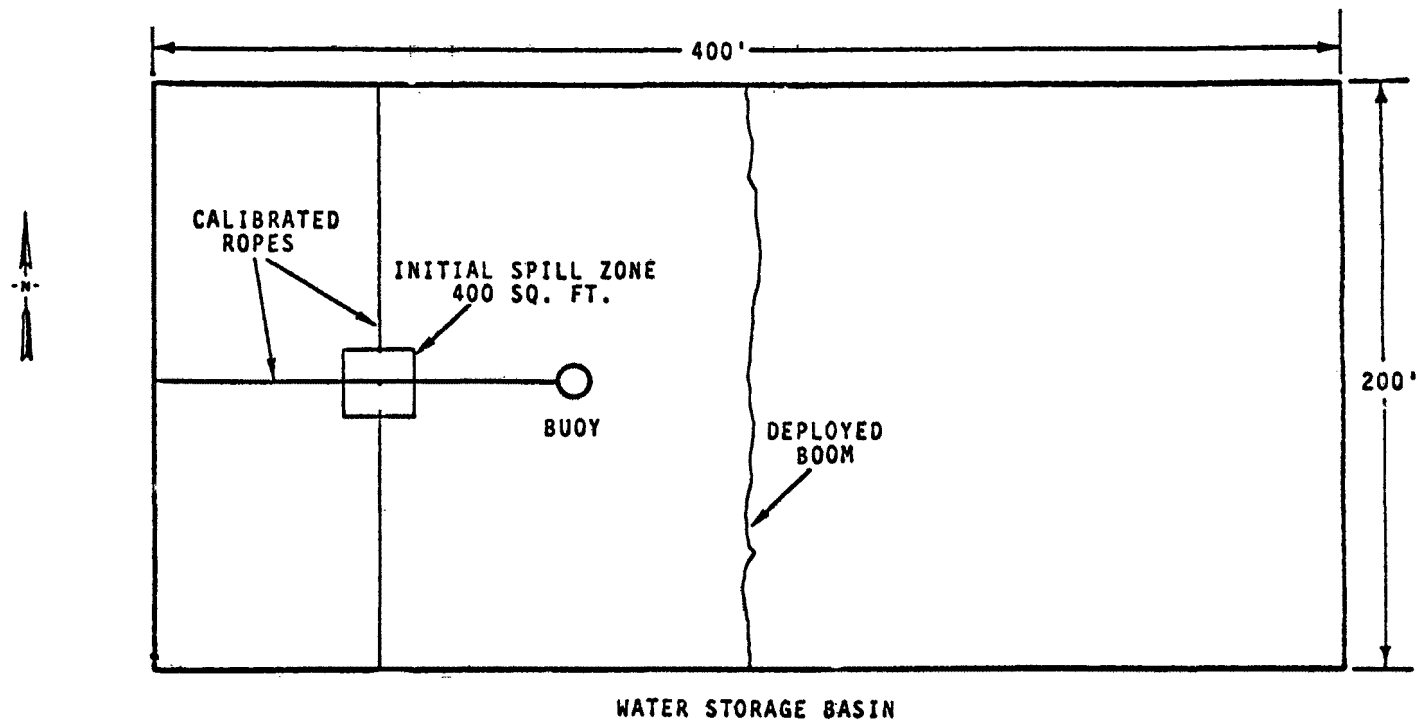
FIGURE 8. Location of facilities for Diazinon spill.

TEST DESCRIPTION

The Diazinon was spilled at 1000 hours by spreading the 96 ℓ (25 gal) of emulsion from a rowboat in a 6.1-m by 6.1-m (20-ft by 20-ft) square as illustrated in Figure 8. Sampling of the spill area began at 1030 hours. Samples were taken with 3-m (10-ft) aluminum tubes connected by flexible tubing to peristaltic pumps. One-liter glass bottles were filled with water withdrawn from 0.3-, 1.5-, and 3-m (1-, 5-, and 10-ft) depths. Sample lines were thoroughly flushed between sampling points.

A total of 260 kg (574 lb) of Nuchar C-190 activated carbon was employed in treating the spill, which is about a 7:1 ratio of carbon to Diazinon. The carbon included 130 kg (287 lb) of 40 x 325 mesh carbon and 130 kg (287 lb) of 325 mesh carbon.

The carbon was housed in the two aluminum bins described in the previous section. These were kept floating in the water while the barge was positioned over the spill zone. At 11:30 the first tote bin was drawn into the slurry inset and strapped in position while the receiving end of the slurrier was brought up into contact with the bin outlet. An attempt was then made to open the valve, however. The pin set to connect the control lever and the valve itself jammed so that the valve stem could not be rotated. Consequently, the first bin was replaced with the second. When pumping commenced, it was determined that the bin was pressurizing rather than being subjected to vacuum. When the pressure was released from the top of the bin, the pressurized water flowed in, wetting much of the carbon. Subsequent investigations revealed that this malfunction resulted from a kink in the slurry hose, which prevented the slurry from being discharged in the surge tank.

It was not possible to clear the blockage without removing the barge from the basin and redoing the fitting. Since time did not permit this, a change was made in the hosing to permit an alternate mode of operation. The latter consisted of using the delivery (surge tank) pump to supply basin water to the top of the bin, thus wetting the carbon. The slurry pump hoses were then switched so that water was pumped from the bin through the pump and into the water column. This allowed for the evacuation of the bin that was serving as a wetting tank.

Operation in this manner allowed for the wetting and delivery of approximately 260 kg (574 lb) of the C-190 carbon. A black suspension was soon apparent in the water. This quickly began to form a mat on the surface. Even coverage of the spill area was achieved through manipulation of the delivery hose and the barge.

At 1600, a sampling boat was launched and post-spill water sampling was initiated using the same procedure and grid as before.

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TEST RESULTS AND DISCUSSION

The samples were analyzed for both phosphate and total organic carbon (TOC). The latter determination may be subject to some error due to the presence of oil slicks from previous spill studies in the basin water. Phosphate was determined by digesting an aliquot of the sample with a sulfuric acid-nitric acid mixture prior to colorimetric measurement of the phosphate concentration by the ascorbic acid-phosphomolybdate method. Analysis of basin water with known concentrations of Diazinon gave 98% recovery of the phosphate by this procedure (2). The TOC analyses were performed with a Beckman Model 915 carbon analyzer (Figure 9).

Results of the phosphate analysis of the pretreatment and post-treatment samples are given in Figure 10, with the locations on the sample grid. Several increases in phosphate concentrations are noted between pre-treatment and post-treatment samples which are believed to be largely caused by sampling variations. The data illustrate that the emulsion was relatively dense and the bulk of the spilled Diazinon formed a layer near the bottom of the basin. Laboratory studies conducted with the 6-in. diameter column did not indicate bulk movement of a fine emulsion to the bottom of the column (2). The density of the Diazinon is greater than water while the density of the xylene in the emulsifiable solution is less than water. Large droplets of the emulsifiable solution will float when initially dropped in water but will slowly sink after a few minutes. It is postulated that the lighter xylene is either evaporated or extracted into the water causing the density of the droplets to increase.

Observations indicated that, while the emulsion was near the surface, the wind drifted the emulsion briefly to the north and west. However, once the emulsion settled several inches into the water it appeared that the wind had little effect on the position of the plume.

Review of the removal levels indicated a relative removal (post to pretreatment basis) of 76% using phosphate analysis, and 84% using TOC analysis. If residual levels are extropolated over the affected water volume to determine overall removal (residual to amount spilled), phosphate data yield a residual of 1.3 kg (2.8 lb) Diazinon (3.4%) and TOC data yield a residual 0.8 kg (1.7 lb) Diazinon (2.1%). Hence, the combined effect of dispersion, hydrolysis, and treatment is removal of roughly 97% to 98% of the Diazinon. A comparison of pertinent data for this run and for the previous trial with aerial delivery can be found in Table 14.

It is apparent from the data that much less of the Diazinon can be accounted for in the slurry trial than was previously accounted for during the aerial delivery trial. No explanation for this has been developed to date. It is possible that the Diazinon spread beyond the sampling grid in significant amounts, but this level of mobility has not been observed previously during the field and laboratory studies. The post-treatment phosphate data indicate that some of the emulsion plume sank to the bottom and moved eastward. This is not duplicated by the TOC data, which raises some questions about its significance. This, however, was not apparent prior to treatment and probably results from a current set up when pumping was initiated.

30

```
                                    ┌──────────────┐
                                    │ 40 Ft N      │
                                    │ A(35-0)100   │
                                    │ B(3-2)33     │
                                    │ C(5-0)100    │
                                    └──────────────┘

                                    ┌──────────────┐
                                    │ 25 Ft N      │
                                    │ A(6-0)100    │
                                    │ B(3-1)67     │
                                    │ C(1.5-2) 33  │
                                    └──────────────┘

                                    ┌──────────────┐
                                    │ 15 Ft N      │
                                    │ A(6-.5)92    │
                                    │ B(3.5-1.5)57 │
                                    │ C(1-35)-250  │
                                    └──────────────┘

                                    ┌──────────────┐
                                    │ 5 Ft N       │
                                    │ A(6-.5)92    │
                                    │ B(11.5-0)100 │
                                    │ C(2-5)-250   │
                                    └──────────────┘
```

30 Ft W	20 Ft W	10 Ft W	Center	5 Ft E	15 Ft E	25 Ft E
A(2-0)100	A(4.5-0)100	A(9-0)100	A(4-0)100	A(3.5-0)100	A(4-0)100	A(5-0)100
B(1.5-0)100	B(8.5-0)100	B(8.5-0)100	B(20-0)100	B(7.5-0)100	B(3.5-0)100	B(4-0)100
C(1-1)0	C(13.5-.5)96	C(2-3)-50	C(3.5-1.5)57	C(1.5-1.5)-50	C(4-2)50	C(4-2)50

```
                                    ┌──────────────┐
                                    │ 5 Ft S       │
                                    │ A(2-0)100    │
                                    │ B(1.5-1)33   │
                                    │ C(1.5-0)100  │
                                    └──────────────┘

       ┌──────────────┐            ┌──────────────┐
       │      1       │            │ 15 Ft S      │
       │ A ( 2 - 3 ) 4│            │ A(0.5-0)100  │
       │ B            │            │ B(0.5-.5)0   │
       │ C            │            │ C(0.5-.5)96  │
       └──────────────┘            └──────────────┘
```

A 1 Ft Depth

B 5 Ft Depth

C 10 Ft Depth

1 Distance and Direction From C

2 Pretreatment TOC Concentration, mg/l

3 Posttreatment TOC Concentration, mg/l

4 Percent Reduction

```
                                    ┌──────────────┐
                                    │ 25 Ft S      │
                                    │ A(2.5-0)100  │
                                    │ B(3.5-0.5)96 │
                                    │ C(3-1.5)50   │
                                    └──────────────┘
```

FIGURE 9. Results of TOC analysis on pretreatment
          and post-treatment samples.

40 Ft N
A(.032-.030)6
B(.188-.230)-20.
C(.126-0)100

25 Ft N
A(2.100-.044)98
B(.346-.240)30
C(.330-.734)-200

15 Ft N
A(2.120-.002)99
B(.420-.230)48
C(.074-1.224)-1000

5 Ft N
A(1.575-.010)99
B(.844-.020)97
C(.015-1.900)

30 Ft W
A(.240)
B(.104)
C(.188)

20 Ft W
A(.404-0)100
B(3.250-.096)99
C(1.600-.710)55

10 Ft W
A(.550-0)100
B(2.940-0)100
C(.170-.642)-100

Center
A(.090-0)100
B(6.090-.074)99
C(.018-.130)-1000

5 Ft E
A(.040-0)100
B(1.130-0)100
C(.120-.630)-500

15 Ft E
A(.086-0)100
B(0.84-0)100
C(.122-.778)-1000

25 Ft E
A(.074-0
B(.170-0
C(.130-6

5 Ft S
A(.084-.046)50
B(.606-0)100
C(.200-.002)99

1
A (2 - 3) 4
B
C

A 1 Ft Depth

B 5 Ft Depth

C 10 Ft Depth

1 Distance and Direction from Center

2 Pretreatment PO$_4$ Concentration, mg/l

3 Posttreatment PO$_4$ Concentration, mg/l

4 Percent Reduction

15 Ft S
A(.038-0)100
B(.290-0)100
C(.100-.078)12

25 Ft S
A(.044-0)100
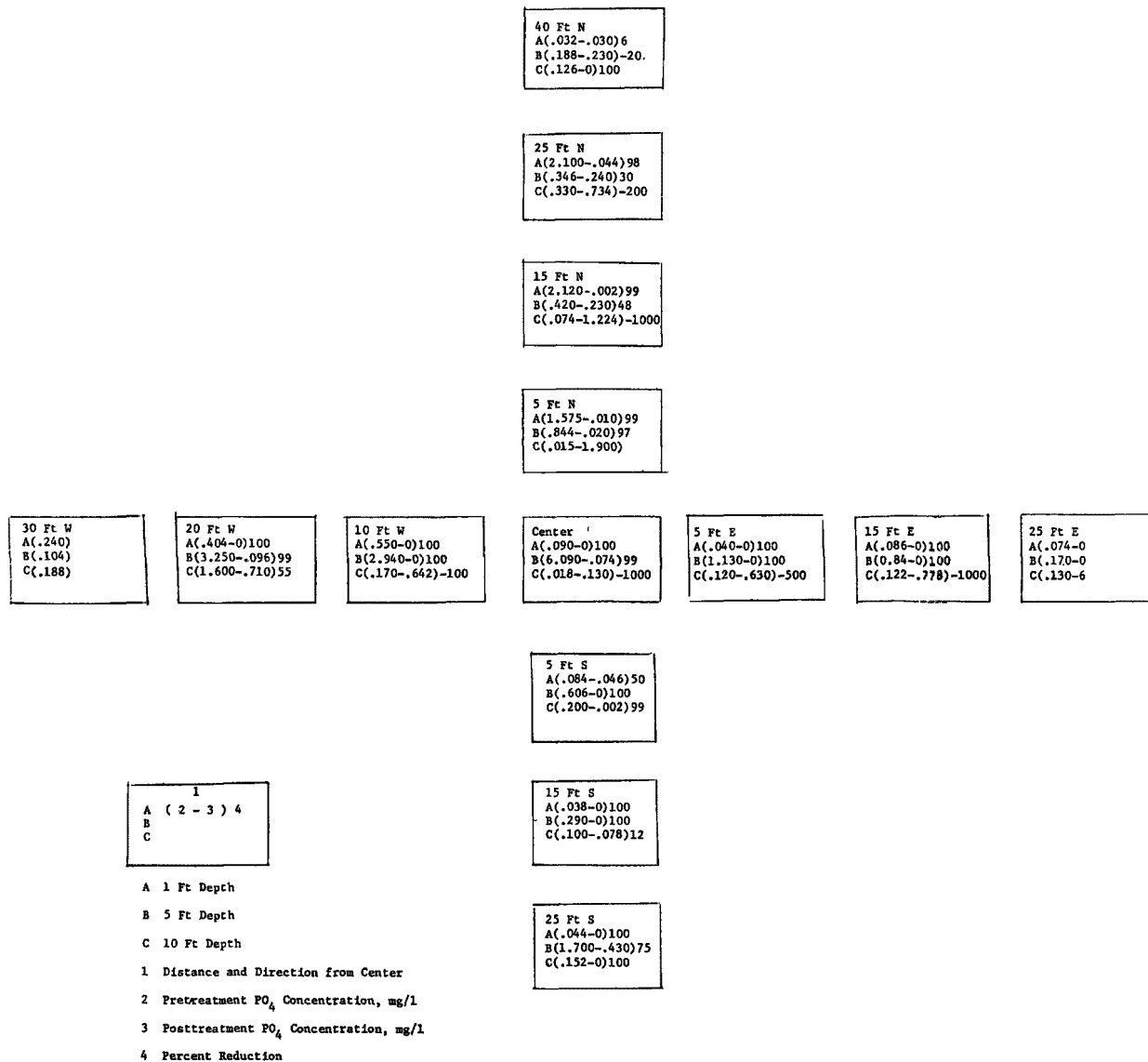B(1.700-.430)75
C(.152-0)100

FIGURE 10. Results of phosphate analysis on pretreatment
            and post-treatment samples.

TABLE 14. COMPARISON OF AERIAL APPLICATION AND SUBSURFACE SLURRY INJECTION

	Dose Level Carbon:Diazinon	Relative Removal (%) (Ave.Post-Treatment) (Ave.Pretreatment)	Overall Removal (%) (Residual Diazinon) (Amt Spilled)	Amount Accounted for Pretreatment (Samples/Amt Spilled) (%)
Aerial Delivery Phosphates (mg/ℓ)	10:1	93.8	95	68
Slurry Delivery Phosphates (mg/ℓ)	7.5:1	76	96.6	11
Aerial Delivery TOC (mg/ℓ)	10:1	92.4	94.5	85
Slurry Delivery	7.5:1	84	97.9	24

Retrieval of spent media was accomplished with a skirted oil boom (Figure 11). No quantitative data were collected on this phase of the trial since collection was documented in earlier studies with serial delivery. Details on performance can be found in Mercer et al. (2).

Despite the operational difficulties encountered with the prototype in the field trial, the use of a slurry dispersion system appears to be effective. Removal efficiencies approached those for the serial delivery trial. Consequently, pursuit of the slurry delivery approach appears warranted. Observations made during the trial should lead to a more flexible design as noted previously.

The slurry approach appears particularly well suited for deployment in two modes, namely: on barges in port areas with a high spill frequency, and as permanent in-stream facilities downstream from major spill sources. The former approach could incorporate the dispersed system with skimmer devices and utilize carbon reuse to maximize efficiency. The latter approach could operate somewhat along the lines of a soaker sprinkler, creating upon demand a screen of carbon across the width of the river.

Regardless of the specific applications, the utility of all these approaches rests on the availability of buoyant carbon. While a means for producing buoyant carbon from available materials has been demonstrated, it is an expensive and unique product that no firms are presently prepared to produce. The future of buoyant media technology is tied closely to the emergence of a reliable, commercial source.
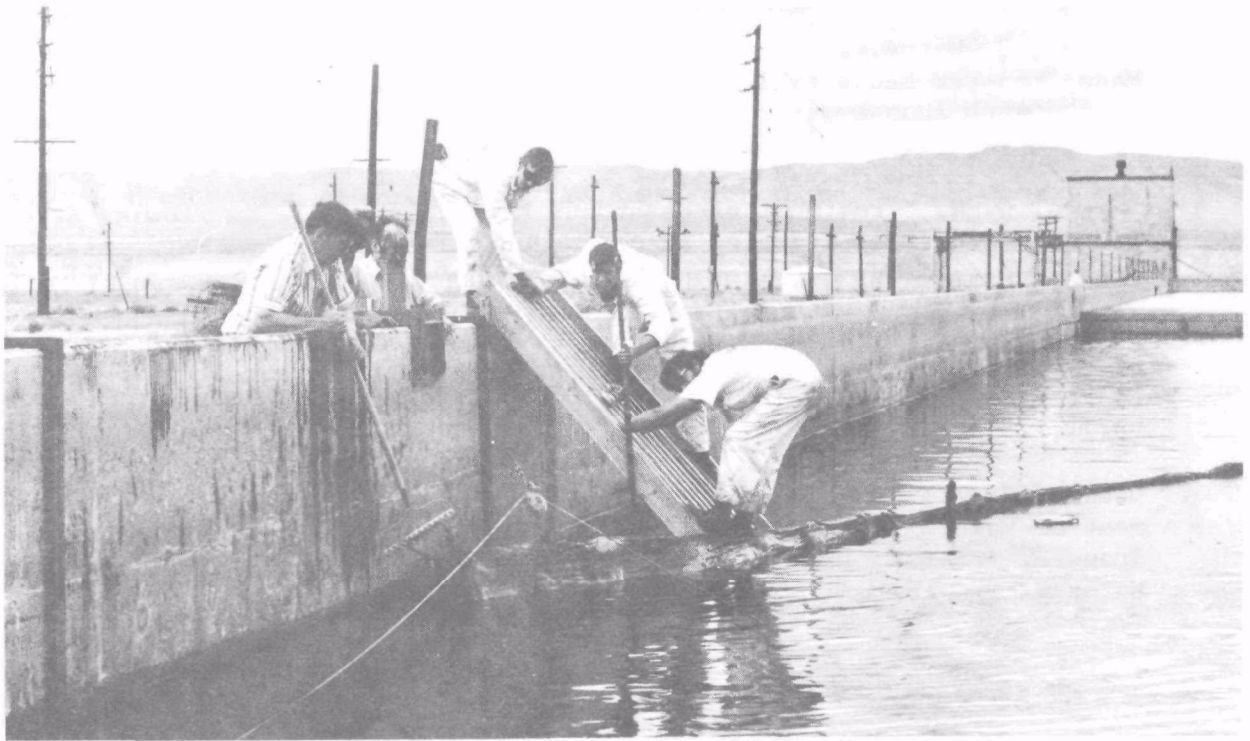
FIGURE 11. Booming.

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16. ABSTRACT

    A prototype system was designed and developed to slurry buoyant activated carbon into a static body of water. The process was developed to remove spilled soluble hazardous compounds from a watercourse. In a simulated spill, up to 98% removal of Diazinon, an organophosphorus pesticide, was achieved by adsorption on activated carbon and by dispersion of the spilled material.

    The basic system was barge-mounted with an intake pump, a jet-slurrier, a surge tank, and a slurry pump. The buoyant carbon was fed into the slurrier by gravity from a floating, hopper-bottom tote bin.

    Since no acceptable buoyant activated carbon is commercially produced in the United States at this time, a method of making buoyant activated carbon by using microballons and a carbon coating mix was developed. Estimated cost per pound of media was $3.50 on a small-batch basis.

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