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Sampling Oil-Water Mixtures at OHMSETT (Oil and Hazardous Materials Simulated Environmental Test Tank)

Mason and Hanger-Silas Mason Co., Inc.
Leonardo, NJ

Prepared for

Environmental Protection Agency, Cincinnati, OH

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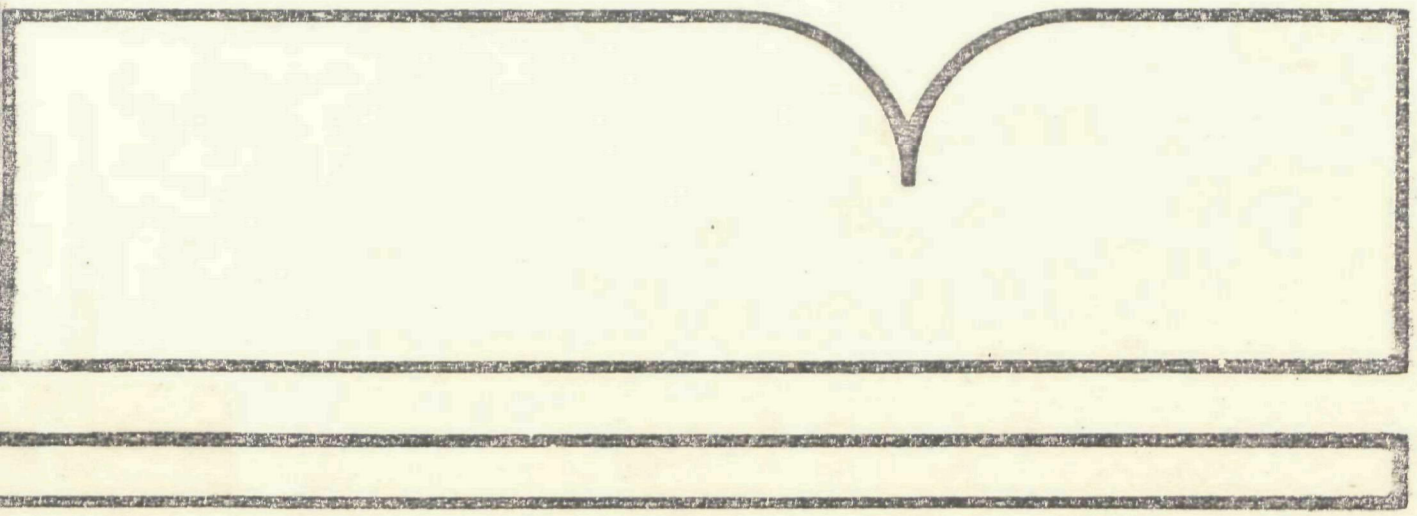

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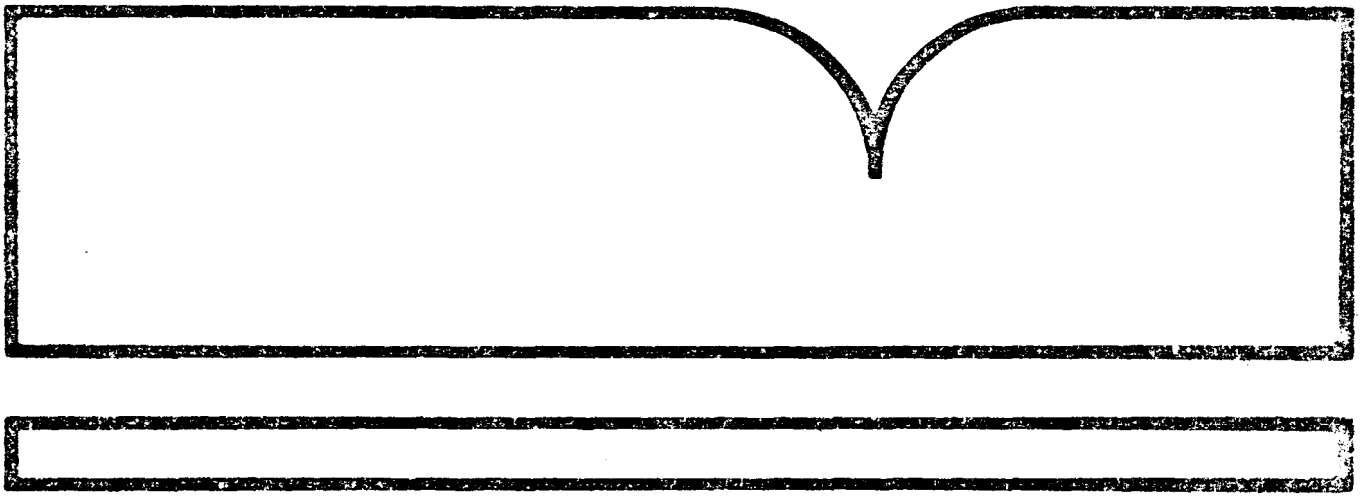

EPA/600/2-87/073
September 1987

SAMPLING OIL-WATER MIXTURES AT CHMCETT

PB88-102892

by

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Contract No. 68-03-3056

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U.S. ENVIRONMENTAL PROTECTION AGENCY
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This report describes procedures developed at OHMSETT for sampling oil and water mixtures.

Two procedures for sampling in containers are discussed: grab and stratified sampling. Both of these techniques require stripping free-standing water from the container bottom. The grab sample technique requires that the remaining fluids be thoroughly mixed before immersing a bottle through the resulting homogeneous emulsion. The stratified sampling procedure uses a sample thief to capture a segmented cross-section of the remaining fluids.

Two procedures for sampling flowing fluids were tested. The two sampling tubes tested were installed immediately downstream of a series of static mixers and a centrifugal pump. The sampling ports were a simple slotted tube and a pilot-shaped tube.

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FOREWORD

As hazardous waste continues to be one of the more prominent environmental concerns to the people of the United States and other countries throughout the world, there are continuing needs for research to characterize problems and develop and evaluate alternatives to addressing those problems. The programs of the Hazardous Waste Engineering Research Laboratory (HWERL) are designed to contribute to satisfying these research needs.

This report describes procedures developed at OHMSETT for sampling mixtures of Circo Medium test oil (a refined naphthenic oil) and Sandy Hook Bay water (salt content approximately 21 ppt). It includes a grab sample technique, a stratified sampler and several discrete sampling procedures. The accuracy and applicability of each technique is addressed. This report is submitted as partial completion of Contract No. 68-03-3056, Job Order 75, which was sponsored by the U.S. EPA. Further information may be obtained through the Releases Control Branch of the Hazardous Waste Engineering Research Laboratory, Edison, New Jersey 08837.

Thomas R. Hauser, Director
Hazardous Waste Engineering Research Laboratory

ABSTRACT

This report describes procedures developed at CHMGRT for sampling oil and water mixtures. The results of testing done to evaluate each sampling technique are presented.

Two procedures for sampling in containers are discussed: grab and stratified sampling. Both of these techniques require stripping free-standing water from the container bottom. The grab sample technique requires that the remaining fluids be thoroughly mixed before immersing a bottle through the resulting homogeneous emulsion. The stratified sampling procedure uses a sample thief to capture a segmented cross-section of the remaining fluids. The grab sample results proved to be within 3% of the known relative oil content in all tests that were considered valid. The grab sample results averaged less than 2% different from the known oil content over the range of 9 to 91% oil. The stratified sampler was tested using several procedures. In the most complete analysis, the stratified sampler gave results within 3% of the known oil content, averaging less than 2% different.

Two procedures for sampling flowing fluids were tested. The two sampling tubes tested were installed immediately downstream of a series of static mixers and a centrifugal pump. The sampling ports were a simple slotted tube and a pitot-shaped tube. The slotted tube results were always within 8% of the grab sample analysis and averaged less than 4% different from the grab sample analysis in all cases. The pitot-shaped tube gave results within 7% in each case and averaged within 4% of the grab sample analysis.

This work is submitted by Mason & Hanger-Silas Mason Co., Inc. as partial completion of Contract No. 68-03-3056, Job Order 75 which was sponsored by the U.S. Environmental Protection Agency. Further information may be obtained through the Releases Control Branch of the Hazardous Waste Engineering Research Laboratory, Edison, New Jersey 08837.

CONTENTS

FIGURES

TABLES

SECTION 1

INTRODUCTION

The U.S. Environmental Protection Agency owns the Oil and Hazardous Materials Simulated Environmental Test Tank (OHMSETT) located in Leonardo, New Jersey. Since its inception, OHMSETT has tested oil spill recovery and control devices in an environmentally safe manner. The daily dealings with oil and water have led to the development of sampling techniques for sampling mixtures of these two immiscible fluids. The need to know with reasonable accuracy the relative proportions of oil and water in a container required that a consistent means of sampling and quantifying the oil content in containers and in flowing streams be developed.

The procedures used at OHMSETT to measure the relative oil-water content in a tank have developed since the facility was first operational in 1974. The earliest measure of the oil percentage in a container was idealistic. A tape measure was used to measure the total height of collected fluids and the height of the oil portion alone. The ratio of the height of the oil portion to the total fluid height was considered the relative oil percentage in the tank. Experience dictated that improvements were necessary. The first attempt to improve this measurement involved stripping free-standing water before taking the second height measurement. This procedural change reduced the error introduced by misreading the oil/water interface due to an oil coating on the sides of the container. The next change accounted for water caught up in the oil after stripping. A bottle was lowered through the remaining oil-rich liquid. The sample was analyzed according to procedures outlined by the American Society for Testing and Materials (ASTM) Procedure 1796.

The work described in this report began with another change that was implemented to improve the quality of the sample of the oil-rich liquid. This procedure involves mixing the oil and remaining water to produce a homogeneous emulsion of the oil-rich layer for sampling. Grab-sampling was still used and is the first technique discussed here. The method of sampling used most frequently during the testing of oil spill control and removal equipment has been the grab sample technique. A second technique was developed to avoid the intentional emulsification of the test oil by using a stratified sampler known as the Johnson Sampler. A third technique used to sample mixed fluids flowing within a pipe or hose, is used at OHMSETT when a real-time analysis is required. This technique uses a small diameter pipe to divert a portion of the flowing mixture to a container for analysis. The fourth technique employed is simply taking a cup and snatching a portion of an open-air flow as the mixture free falls from the end of the carrier. This last technique has been used infrequently, and as a last

resort when only rough estimates of oil content were required and there was no other means of obtaining the desired sample. The most common sampling need during OHMSETT tests has been sampling oil and water in a container. Therefore, grab samples and stratified samples have been most commonly taken.

SECTION 2

CONCLUSIONS

Mixtures of oil and water can be sampled to reflect accurately the relative oil/water content of either tanks or flowing streams. The sampling technique that is chosen for use is dependent on the type of process involved (i.e. batch or dynamic) and available resources.

SAMPLING FROM CONTAINERS

Either the stratified sampler or grab sampler can be used to obtain a representative sample of the oil-water mixture in a container. It is important to realize that as much of the water phase as is possible should be removed before sampling because of the techniques used in the laboratory analysis. The pear shape of the centrifuge tube allows for highly accurate measurements of low water-content samples (less than 2% water) but introduces a significantly greater measurement error in high water-content (greater than 25% water) measurements. The grab sample technique will give measurements within 3% oil of the actual oil content of the fluids in a container. The stratified sampling technique gives measurements within 3% of the actual relative oil content. Shortcuts will give measurements within 15%, depending on which shortcut is used.

The stratified sampler, when constructed with a transparent sheath, provides a visual cross-section of the materials and layers within the tank. This would be of value when an immediate estimate of fluid quality is needed. This is, of course, only feasible when the sheath is constructed of a material chemically compatible with the sampled material or the general nature and reactivity of the material to be sampled is known.

These tests were conducted using only OHMSETT's Circo Medium test oil and Sandy Hook Bay water. The results can presumably be replicated using many other fluids. If more than one liquid is present, and the liquids are not mutually completely soluble, the stratified sampler will not give a representative sample in irregularly shaped tanks. In such a case, the grab sample technique would be preferable.

The major drawback of the grab sample method is the need to mix the liquids thoroughly. This extends the time required for sampling, requires that power be available to make mixing feasible, and hinders later separation of the liquids.

Neither of these two techniques is readily applicable to inflatable tank or bladder type storage, but stratified sampling would most likely

give better results in this type container provided that depth-volume estimates could be established.

SAMPLING FROM FLOWS

The pitot-shaped-tube and the slotted-tube sampling ports gave an equally representative sample of the flowing fluid when used in combination with the static mixers. The sampling tubes gave estimated differences of 5% from the grab sample analysis of the total fluid pumped for the pitot tube and slotted tube.

The dispersion produced by the static mixers readily coalesced in the receiving barrel, demonstrating that the mixing of the oil with the water was only a temporary condition. This further demonstrates that the sampling port must be located immediately downstream of the mixers to avoid coalescence within the carrying pipe or hose.

SECTION 3

RECOMMENDATIONS

When sampling oil-water mixtures in containers, stratified samplers should be used whenever the container is constructed with vertical, parallel sides. The time period allowed for gravity separation of the immiscible fluids should be as long as practical so that the container can be readily stripped of free-standing water.

In other sampling applications, grab samples should be taken following the procedures outlined. A thorough mixing is necessary to create a homogeneous emulsion for sampling.

When it is necessary to sample flowing mixtures of oil and water, either the pitot tube-shaped sampling port or the slotted-tube sampling port can be used with equal accuracy. Whichever sampling port is chosen, it should be located immediately downstream of the static mixer. The choice of sampling port will be dependent on the specifics of the case involved and the fabrication capabilities available. When used in the field, several of both types should be readily available from a supply established prior to the spill.

Each of the sampling techniques that was tested used only OHMSETT's Circo Medium test oil and salt water. If these techniques are to be used with different fluids from these, a wide variety of mixtures should be investigated to ensure that the physical properties of the fluids involved do not adversely affect the devices. The principal areas that need to be investigated further are the effects of viscosity, specific gravity, surface tension, and mutual immiscibility. Although it is anticipated that these results will be applicable for a wide range of mixtures, there may be cases where these devices do not perform equally well. For example, the stratified sample thief may not be able to capture a sample in a highly viscous fluid. If the fluid does not flow into the annular space during the time that the sheath is removed, there will not be a sample taken. It is equally likely that the continuous phase of the emulsion will selectively flow into the annular space giving a non-representative sample. Similarly, if the flowing fluids have high content of stringy fibers, static mixers may plug quickly, leading to excessively large pressure drops, making this approach to sampling impractical.

Another extension of this work should be a repeatability and confidence-level investigation to confirm statistically the error of each method. It may be necessary to use only one of the test fluids unless the physical properties prove be of significant influence.

SECTION 4

SAMPLING FROM CONTAINERS

When evaluating an oil skimmer, a mixture of oil and water has been typically pumped into a collection container from the skimmer. In order to determine the recovery efficiency, throughput efficiency, and oil recovery rate, the volumes of oil and water in the collection tank or tanks had to be determined.

These containers have traditionally been free-standing with vertical parallel sides so that the volume was directly proportional to the height within the container. When containers were used that had variable cross-sectional area, a height-volume calibration was conducted so that volume could be determined directly from fluid height measurements.

GRAB SAMPLES

Each time a grab sample was taken, the height of the total collected fluids was measured using a tape measure. This initial height was recorded. The collected fluids were then stripped of as much free-standing water as possible using a valved spigot on the bottom of the tank. When oil began to appear in the stripped water, the valve was partially closed. The stripping process was continued at a reduced rate until the maximum possible amount of water had been removed. The remaining high-oil-content fluids were emulsified for five minutes using a Lightning electric mixer. A glass 100-ml bottle clamped to a rod was slowly lowered through the homogeneous fluid from top to bottom and slowly raised out of the emulsion. The bottle was capped for analysis later in the day. These samples of oil and water were analyzed in accordance with ASTM D-1796.

Test Procedures

Twenty-two tests were conducted by pumping oil into a 1.9-m^3 flat-bottom polypropylene barrel and adding bay water to produce mixtures of oil and water ranging from 9% oil to 91% oil. The polypropylene barrel was cylindrical, 1.2-m in diameter and 1.5-m high. The oil used was analyzed and determined to contain less than 0.1% bottom solids and water, which was assumed to be negligible. The height of the oil in the tank was measured using a tape measure and sighting the height of the fluid through the translucent tank walls and recorded. The test oil had a viscosity of less than 1,000 cSt at the ambient temperature. The height of the combined oil and water in the tank was also measured and recorded. These two tank soundings were used to determine the true oil percentage in the barrel. The expected error in these two measurements yield an error in oil content calculation of less than 1% oil.

The fluids in the containers were agitated slightly with the mixer and sampled as outlined. The percent oil in the container was calculated by multiplying the volume after stripping by the percent oil in the grab sample as determined by laboratory analysis, dividing by the initial volume, and multiplying by 100.

Results

Of the 22 tests, 20 were considered valid. Two runs are discounted because the height of the fluid remaining after stripping was insufficient for proper mixing, therefore the homogeneity of the fluid sampled is suspect. A least-squares linear regression on both variables for all 22 points yields:

$$y = 1.00 \times +1.61 \qquad r = 0.993$$

A second regression with the two data points discounted yields:

$$y = 0.97 \times +2.03 \qquad r = 0.996$$

In both cases, y is the percent oil in the tank as analyzed, x is the known percent oil by volume, and r is the correlation coefficient for the regression. The data are listed in Table 1 and graphed in Figure 1. The 20-point results showed that the oil content averaged less than 2% oil different from the known oil content. The 95% confidence limit showed that the difference should not be expected to exceed 3% oil (see Appendix B, Table B-1).

The oil content determined should be expected to be higher than the actual oil content. Both the regressive analysis and the student t-test show the bias. It is believed that the bias is introduced by two factors. First, the electric mixer does not produce a truly homogeneous emulsion. The container originally contained a higher oil-rich phase and the resulting emulsion also contained a higher oil content at the top than on the bottom. The second cause is the insertion method of the 100-ml bottle. The bottle first samples the higher layers of the container. If the bottle is filled prior to reaching the bottom of the container, then the lower level will not be sampled. The combination of these are systematic to producing a higher-than-true oil determination. This is further supported by the excluded tests. Observation showed that the tank was improperly mixed. The fluids were known to be nonhomogeneous. The resulting analysis showed much higher than known oil content.

The statistical analysis shows that the points excluded are statistical outliers. This supports ignoring them in additional analysis. The third data point is also a statistical outlier. It has not been discarded because there was no observed discrepancy in the test.

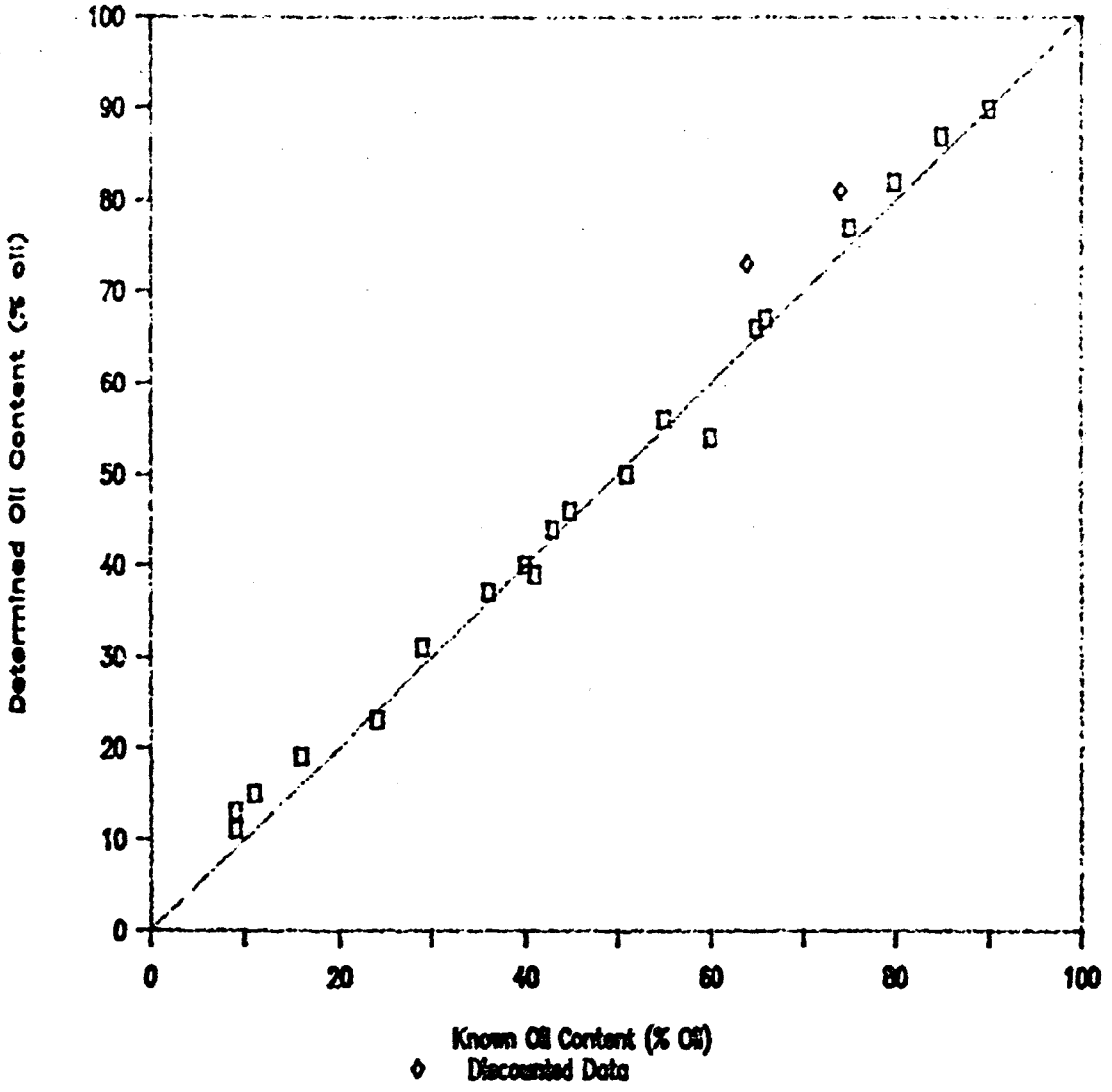

Figure 1. Results of grab sample testing. Note that the line drawn shows perfect correlation and is not the results of curve fitting.

TABLE 1. GRAB SAMPLE QUALITY TESTS (22 Tests)

Known Oil Content (% oil)	Determined Oil Content (% oil)	Difference (% oil)	Absolute Difference (% oil)
74	81*	-7	7
64	73*	-9	9
60	54	6	6
66	67	-1	1
45	46	-1	1
55	56	-1	1
75	77	-2	2
85	87	-2	2
65	66	-1	1
80	82	-2	2
40	40	0	0
51	50	1	1
43	44	-1	1
36	37	-1	1
41	39	2	2
29	31	-2	2
24	23	1	1
9	13	-4	4
16	19	-3	3
9	11	-2	2
11	15	-4	4
90	90	0	0

* Tests discounted due to improper mixing caused by low height of stripped fluids in the tank.

STRATIFIED SAMPLING

The second means of sampling tanks containing both oil and water at OHMSETT is using the stratified sample thief known as the Johnson Sampler. The device was developed by OHMSETT Test Director Michael G. Johnson to avoid the intentional emulsification of the test oil, as all of the test oils are refurbished and reused at OHMSETT. This device (see Figure 2) was constructed of three major segments: an inner core, an outer sheath, and a handle. The inner core of the sampler was constructed so that each of the annular segments captured 51 mm of sample height.

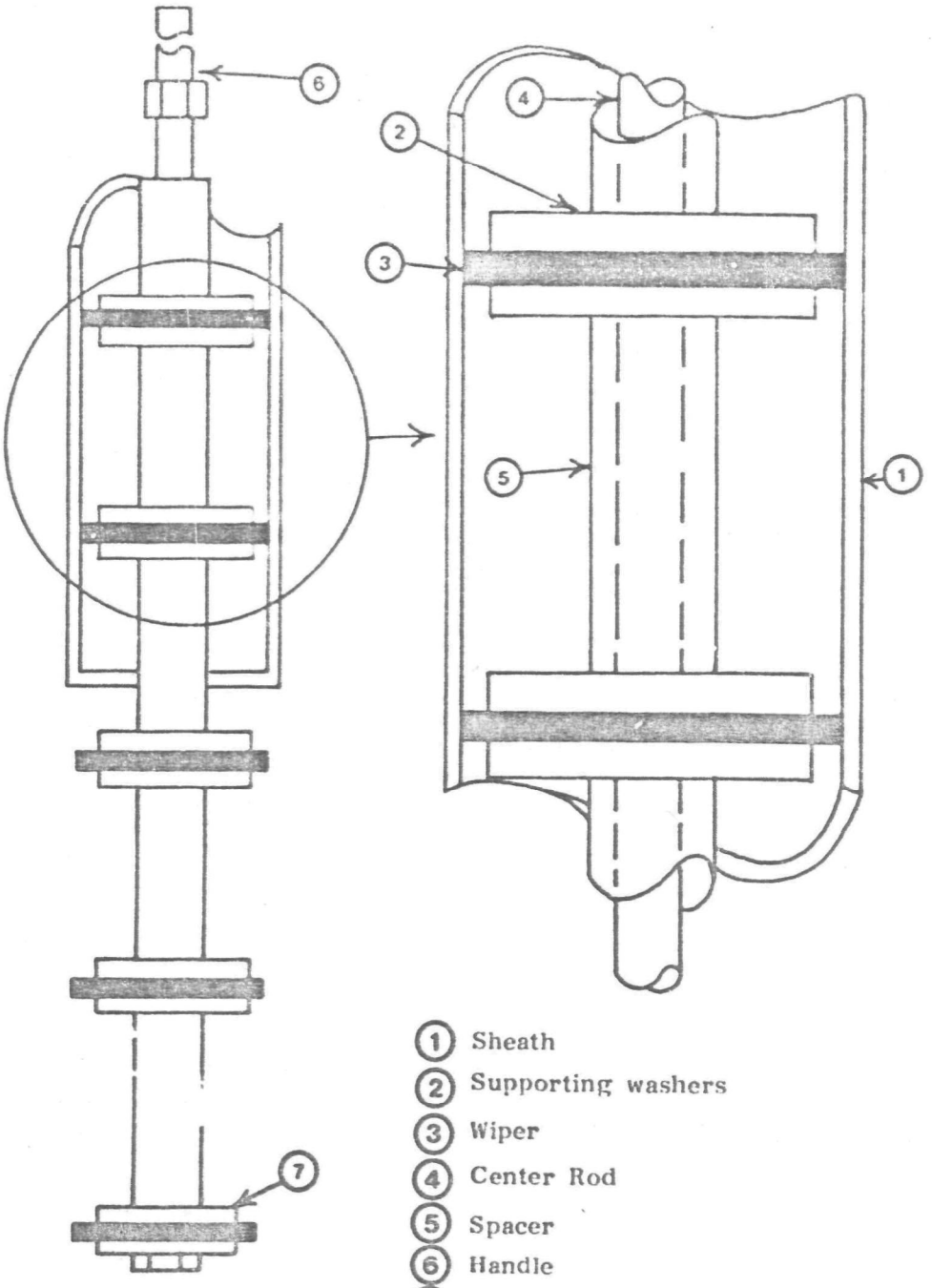

Figure 2. Stratified sampler with construction details

The samplers used at OHMSETT for this study were each 1.2-m long with a 1.2-m handle. The size of the sampler was selected to match the height of the container to be sampled. A sampler that was too short would not have provided a representative sample of the entire tank. An excessively long sampler would have given an equally good sample, but would have been cumbersome. The sampler was used by inserting it into the tank and perpendicular to the fluid surface. The sheath was removed to allow the fluids to flow into the annular spaces. Replacing the sheath trapped the sample. The entire sampler was then removed. The entire stratified sampler, shown in Figure 2, was then taken to the lab for analysis of the fluids.

Tests

The stratified sampler was tested for accuracy and convenience in essentially the same fashion as the grab sample technique. The containers were sampled after mixing for a short period to simulate passage through a pump, both before and after stripping the free-standing water. During these tests, the fluid collected by the samplers was drained into 100-ml graduated cylinders through a funnel and allowed to settle for between 2 hours and 3 days in an attempt to find a field-worthy alternative to following the complete ASTM D-1796 standards. The need to strip all free-standing water before sampling was investigated. The combination of not stripping the excess water and separating in the graduated cylinder analysis was also investigated to determine the accuracy of the sampling and analysis technique should a case arise where no outside power supply was available to drive the centrifuge for a more complete analysis, or where stripping of the bottom layer was not convenient. This would be the case when the content of the heavier fluid is desired or the fluids could not be visually distinguished. The stratified sampling thief was, therefore, tested using four methods. Method 1 was the most complete analysis. The container was stripped of free-standing water prior to taking the sample, and the captured liquid was analyzed by the ASTM procedure. Method 2 also used the ASTM analysis but the sample was taken prior to stripping. In Method 3, the sample was taken after stripping, but the water content was determined using a graduated cylinder. Method 4 also used the graduated cylinder but the sample was taken prior to stripping the free-standing water.

Results

Method 1, using a stratified sampler and maximum stripping gave an accurate determination of the oil content in the container. The difference between the known and measured oil content of the container was found to be less than 3% oil at the 95% level of confidence. The measured difference was biased lower than the known oil content (see Figure 3 and Appendix B). Method 2, using the stratified sampler without stripping free water gave results within 7% oil. The oil content results tended to be high rather than low as shown in Figure 4.

Method 3, substituting the graduated cylinder analysis for the complete ASTM analysis, yielded a consistently high oil content (see Figure 5) because of the assumption that the oil phase is pure oil. This method

yielded results within 15% of the actual oil content. The oil involved has historically shown a tendency to retain some water after exposure, and 10% water volume would not be exceptional. Arbitrarily estimating that the oil phase contained 10% water will cut the statistical error in this analysis in half. The amount of water retained by the oil should be expected to vary from oil to oil. This would affect the above limits. The addition of the solvent to lower the apparent viscosity of the mixture combined with the enhanced gravity separation of the centrifuge in the ASTM method minimized this problem.

When Methods 2 and 3 were combined, the accuracy of the measurement improved to within 6% of the actual value and gave a slightly low average. The scatter decreased and the correlation of the measured relative oil concentration with the known relative oil concentration in the container improved (see Figure 6). It was felt, however, that this may have been a fortuitous coincidence rather than a predictable trend. These results were named as being the field data points and the technique as Method 4. The same cautions applied to Method 3 should also apply to Method 4.

The results of these tests are shown in Table 2 and Figures 3 through 6. The statistical analysis is given in Appendix B.

The complete analysis shows a bias. The oil content determined from the sample taken with the stratified sample thief was consistently lower than the known oil content in the container. One possible explanation of this bias is that some portion of the oil originally in the sample does not completely drain. This is particularly true of the sampler core. Although long drain times were used, an oil coating undoubtedly remained. The comparative volume of the oil coating may account for the 1 to 3% bias in the results.

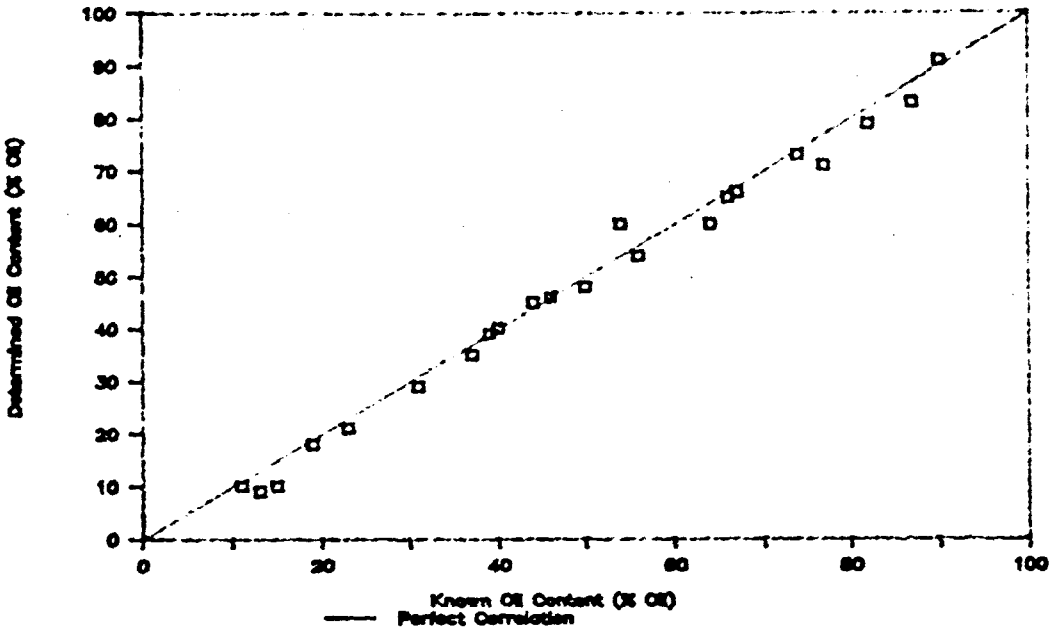

Figure 3. Results of stratified sampler testing using Method 1. Note that the line drawn shows perfect correlation and is not the result of curve fitting.

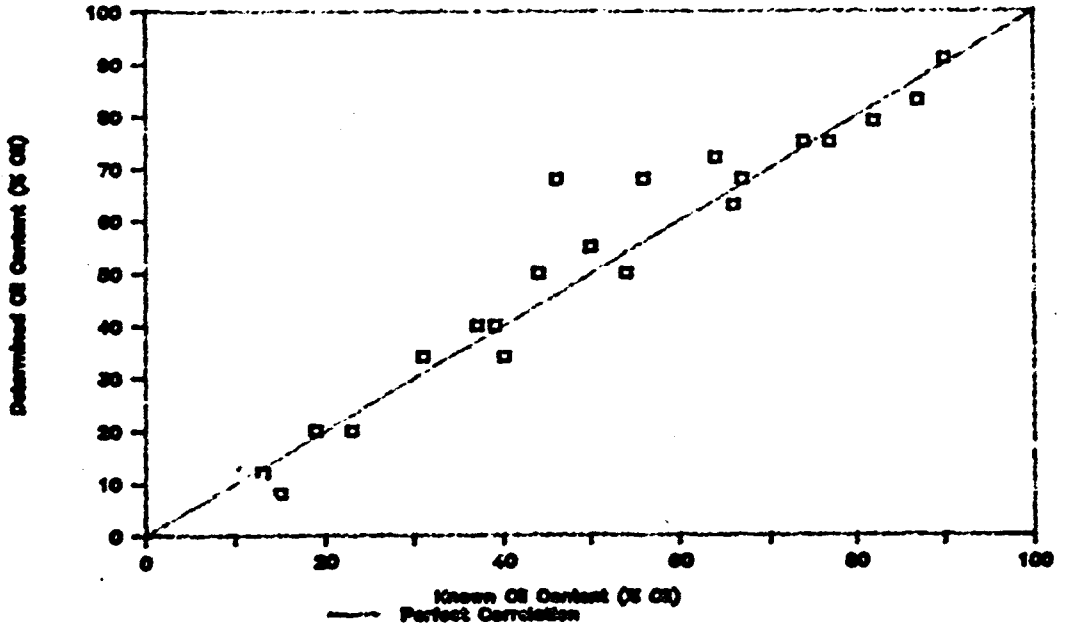

Figure 4. Results of stratified sampler testing using Method 2. Note that the line drawn shows perfect correlation and is not the result of curve fitting.

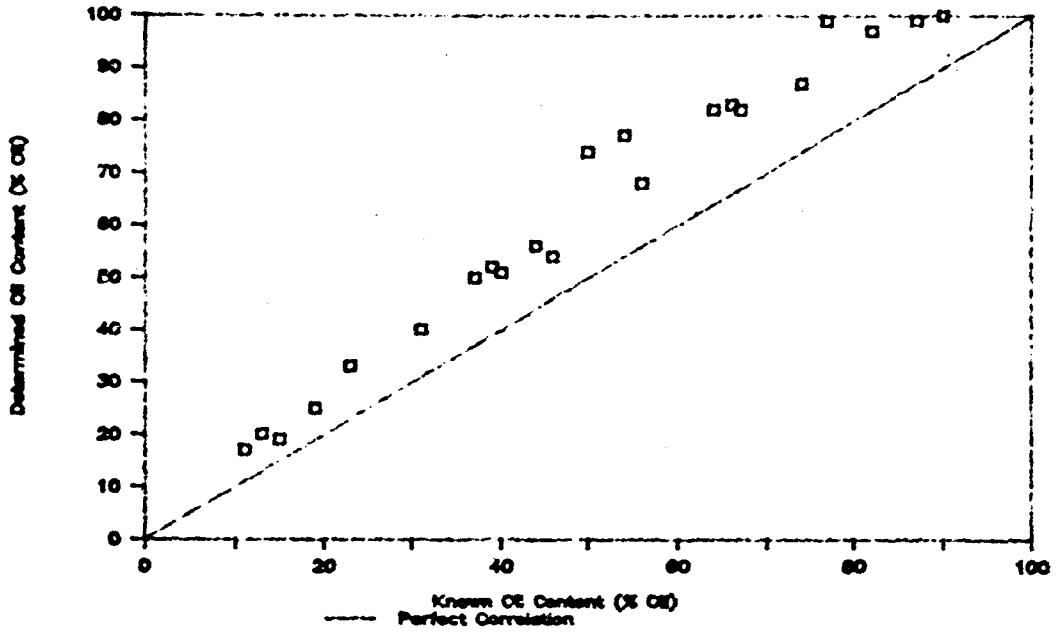

Figure 5. Results of stratified sampler testing using Method 3. Note that the line drawn shows perfect correlation and is not the result of curve fitting.

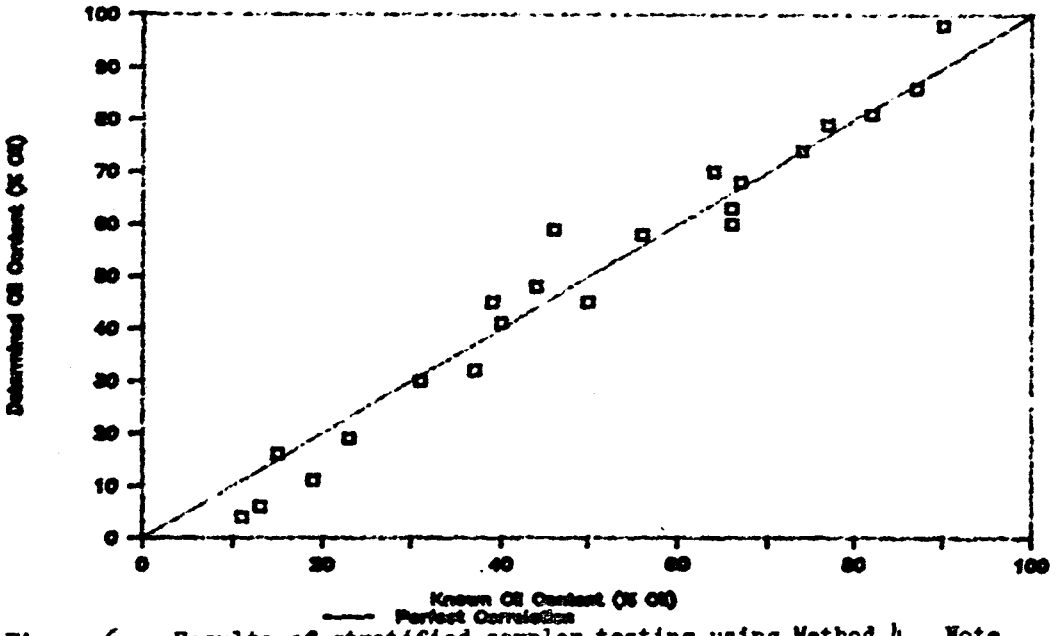

Figure 6. Results of stratified sampler testing using Method 4. Note that the line drawn shows perfect correlation and is not the result of curve fitting.

TABLE 2. STRATIFIED SAMPLERS RESULTS

Known Oil % X	Oil Percent Method Number 1 Y1	2 Y2	3 Y3	4 Y4
74	73	75	87	74
64	60	72	82	70
54	60	50	77	n/a*
67	66	68	82	68
46	46	68	54	59
56	54	68	68	58
77	71	75	99	79
87	83	83	99	86
66	65	63	83	63,60+
82	79	79	97	81
40	40	34	51	41
50	48	55	74	45
44	45	50	56	48
37	35	40	50	32
39	39	40	52	45
31	29	34	40	30
23	21	20	33	19
13	9	12	20	6
19	18	20	25	11
11	10	12	17	4
15	10	8	19	6
90	91	91	100	98

* Sample lost when wind blew over graduated cylinder.
\+ Two samples taken

Linear Regressions

Y1 = 1.001 x - 1.560, r = 0.995
Y2 = 1.004 x + 1.273, r = 0.967
Y3 = 1.124 x + 6.594, r = 0.987
Y4 = 1.109 x - 6.059, r = 0.984

Method 1: Maximum stripping, complete ASTM analysis estimated error, 3% oil
Method 2: No stripping, complete ASTM analysis estimated error, 6% oil
Method 3: Maximum stripping, graduated cylinder analysis estimated error 15% oil
Method 4: No stripping, graduated cylinder analysis estimated error 6% oil

SECTION 5

SAMPLING FROM FLOWS

It has been occasionally desirable to sample oil and water flowing in hoses or pipes when the nature of the flow is unknown. The fluids could be flowing in turbulent or laminar regimes, stratified laterally or radially, or dispersed. It may be at very high or low flow rates. This type of sample has been of particular use when a real-time analysis of the skimmer performance was required or the skimmer evaluation had to be performed on an operating skimmer in an actual field test at a spill of opportunity. There have been three attempts to sample flowing oil and water at OHMSETT. All of the attempts have addressed, to some degree, the nature of two phase flow, a realistic sample size, and ease of fabrication. All three of these techniques have as their basis the interception and diversion of a portion of the flow area to obtain a representative sample of the flowing fluids.

The first device tried was a holed sampler (perforated sampling tube). It was constructed using 6-mm (1/4-inch) pipe with holes of differing sizes drilled at regular intervals. These holes were chosen to be standard twist drill sizes for ease of construction. The second and third devices were a slotted tube and a pitot-shaped tube downstream from a static mixer on the high-pressure side of a pump.

UNMIXED FLOW

When OHMSETT conducted an offshore test of the Shell SOCK skimmer (see Lichte et al., 1981), the discharge of a Tuthill positive-displacement pump was sampled for comparison with the samples taken from the collection tanks. There was no official testing of the holed sampler in this program, but the discrete values of recovery efficiency (percent oil) agreed within about 15% of the values obtained using the tank soundings. Similar sampling ports have been used at OHMSETT prior to the SOCK tests with similar results.

The holed sampler was constructed using 76-mm (3-inch) schedule-40 galvanized steel pipe as the housing to match the pump discharge size. The sampling pipe was 6-mm (1/4-inch) pipe with holes drilled along its length. The hole size and spacing was chosen based on readily available twist drill bit sizes.

MIXED FLOW

A second method of dynamic sampling was tested in an effort to obtain a more representative sample of oil and water flowing together. The heart

of the technique lay in using a static mixer to produce a homogeneous dispersion and creating plug flow conditions within the pipe at the sampling port regardless of the initial flow conditions. It was felt that the completely uniform dispersion in plug flow conditions would make the geometry of the port irrelevant to a great degree.

Two sampling ports were constructed for testing in these trials. The first was shaped like a classic Pitot tube locked into the center of the main flow-carrying pipe. This was generally designed around the specifications outlined by Underwriters' Laboratory specification 1504 but had slight dimensional variations. The second sampling port was constructed using a 6-mm (1/4 inch) pipe with a slot cut the width of a saw blade (approximately 3 mm) along the longitudinal axis.

Static Mixer

The static mixer was chosen over dynamic mixers to eliminate extra power requirements. The mixing energy was provided by the pump. The unit selected for this application was Model X020-040-1-003-22 manufactured by Komax Systems, Inc., Long Beach, California. The mixer was chosen to provide a dispersion of 1000 micron (1 mm), or approximately 20% of the slot width. This size of the dispersion was chosen so that the oil would coalesce readily after sampling to avoid any unnecessary emulsification of the fluids. The static mixer used is shown in Figure 7.

Test Procedures

A Barnes Model 12CCG centrifugal pump was fed an oil-water mixture from two separate containers holding pure oil and water from Sandy Hook Bay. The relative percentage of oil was set by the arbitrarily selected positions on the spigot feed valves. The percent oil, by volume, varied from 21% to 97% during the 12 comparison runs. The pump discharge was fitted with the three static mixers in series and the sampling ports. The slotted tube was followed immediately by the pitot tube. Samples were taken at 60-second intervals, with the first 60 seconds of each test being considered slop. The sampling ports were constantly open. The fluid from the ports was routed to a separate container when not filling the 200-ml sample bottles.

Results

Four to ten samples were taken for each test. The analysis of each test was averaged based on the assumption that there was a constant pumping rate during the test. The average oil content found in the discrete samples was then compared with the percent oil determined by taking a grab sample of the total pump discharge, less the 60 seconds of slop.

The slotted-tube sampler and pitot-tube sampler each had an absolute average difference from the grab sample analysis of approximately 3%. The average algebraic difference from the grab sample analysis was less than 1% in each case. The absolute difference is indicative of the inaccuracies of the method, regardless of direction, i.e., a 1% high measurement or a 1%

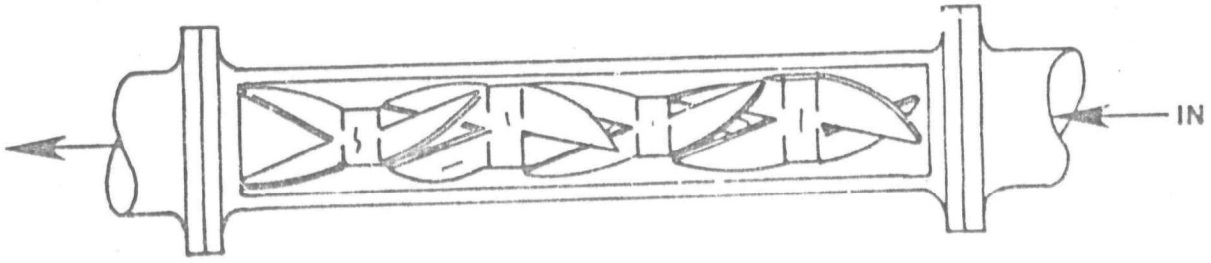

Figure 7. Longitudinal cutaway view of Komax static mixer.

low measurement would give a 1% absolute difference. The algebraic difference incorporates the direction of the difference to reflect the systemic bias of the method. The 95% confidence interval showed the sampling and analysis procedures yielded oil contents within 9% of that found in the grab sample. The data is plotted in Figures 8 and 9. The results are compared in Table 3.

TABLE 3. OIL PERCENTAGES FOR GRAB AND DYNAMIC SAMPLES

Test No.	Grab Sample*1 % Oil	Pitot Tube Average % Oil	Slot tube Average % Oil
A	56	52 (+4)*2	52 (+4)
B	60	58 (+2)	59 (+1)
1	97	97 (+0)	97 (0)
2	86	84 (+2)	84 (+2)
3	78	71 (+7)	73 (+5)
4	75	68 (+7)	73 (+2)
5	41	40 (+1)	46 (-5)
6	36	42 (-6)	41 (-5)
7	29	31 (-2)	36 (-7)
8	28	32 (-4)	31 (-3)
9	21	21 (0)	21 (0)
10	22	28 (-6)	25 (-3)
Average absolute difference		3.4%	3.2
Average algebraic difference		0.4%	-0.8

*1 Post test analysis of the grab sample test results showed that these measurements are within 3% oil of the actual oil content.

*2 Numbers shown in parenthesis are the algebraic difference between grab sample and dynamic samples

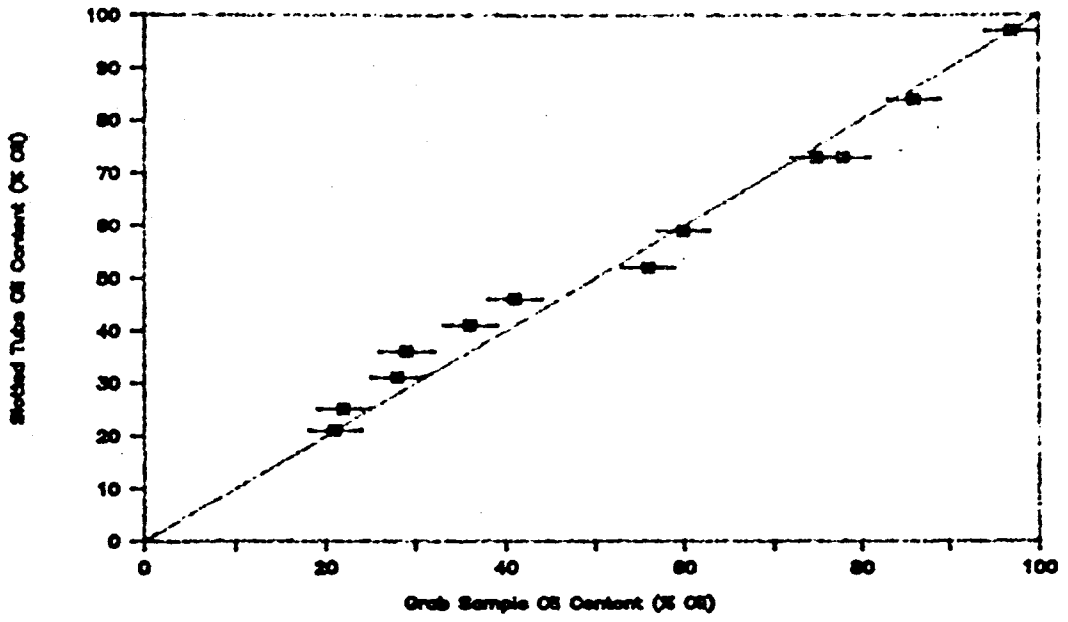

Figure 8. Results of slotted tube tests. Note that the line drawn shows perfect correlation and is not the result of curve fitting.

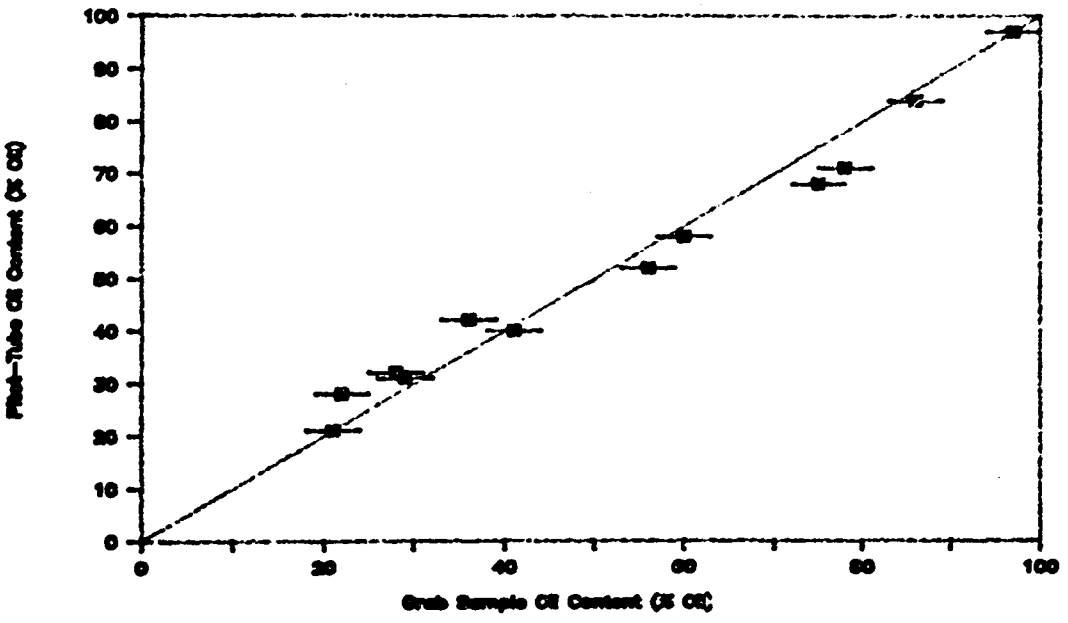

Figure 9. Results of pitot tube tests. Note that the line drawn shows perfect correlation and is not the result of curve fitting.

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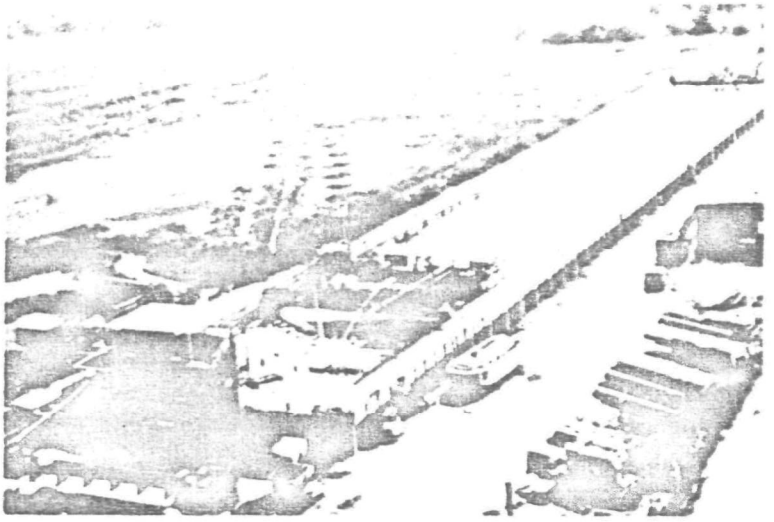
APPENDIX A

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY



The U.S. Environmental Protection Agency operates the Oil and Hazardous Materials Simulated Environmental Test Tank (OHMSETT) located in Leonardo, New Jersey. This facility provides an environmentally safe place to conduct testing and development of devices and techniques for the control and clean-up of oil and hazardous material spills.

The primary feature of the facility is a pile-supported, concrete tank with a water surface 203 meters long by 20 meters wide and with a water depth of 2.4 meters. The tank can be filled with fresh or salt water. The tank is spanned by a bridge capable of exerting a horizontal force up to 151 kilonewtons while towing floating equipment at speeds to 3.3 meters/second (6.5 knots) for at least 40 seconds. Slower speeds yield longer test runs. The towing bridge is equipped to lay oil or hazardous materials on the surface of the water several meters ahead of the device being tested, so that reproducible thicknesses and widths of the test slicks can be achieved with minimum interference by wind.

The principal systems of the tank include a wave generator, a beach, and a filter system. The wave generator and absorber beach can produce regular waves to 0.6 meter high and to 45 meters long, as well as a series of 0.7 meters high reflecting, complex waves meant to simulate the water surface of a harbor. The tank water is clarified by recirculation through a 410 cubic meter/hour diatomaceous earth filter system to permit full use of a sophisticated underwater photography and video imagery system and to remove the hydrocarbons that enter the tank water as a result of testing. The towing bridge has a built-in oil barrier which is used to skim oil to the North end of the tank for cleanup and recycling.

When the tank must be emptied for maintenance purposes, the entire water volume of 9800 cubic meters is filtered and treated until it meets all applicable State and Federal water quality standards before being discharged. Additional specialized treatment may be used whenever hazardous materials are used for tests.

Testing at the facility is served from a 650 square meters building adjacent to the tank. This building houses offices, a quality control laboratory (which is very important since test fluids and tank water are both recycled), a small machine shop, and an equipment preparation area.

This government-owned, contractor-operated facility is available for testing purposes on a cost-reimbursable basis. The operating contractor, Mason & Hanger-Silas Mason Co., Inc., provides a permanent staff of twenty multi-disciplinary personnel. The U.S. Environmental Protection Agency provides expertise in the area of spill control technology and overall project direction.

For additional information, contact: Richard A. Griffiths, OHMSETT Project Officer, U.S. Environmental Protection Agency, Hazardous Waste Engineering Research Laboratory, Releases Control Branch, Edison, New Jersey 08837 Telephone: 201-321-6629.

APPENDIX B

STATISTICAL SUMMARY OF TEST RESULTS

Individual replicates of the tests were not performed as part of this program. There were sufficient individual tests conducted to establish an estimate of overall confidence limits across the oil content range presuming that differences are normally distributed.

TABLE B-1. STATISTICAL SUMMARY OF GRAB SAMPLE TEST RESULTS

OK % Oil	OD % Oil	OK-OD % Oil	OK-OD % Oil
74	81	-7	7*
64	73	-9	9*
60	54	6	6
66	67	-1	1
45	46	-1	1
55	56	-1	1
75	77	-2	2
85	87	-2	2
65	66	-1	1
80	82	-2	2
40	40	0	0
51	50	1	1
43	44	-1	1
36	37	-1	1
41	39	2	2
29	31	-2	2
24	23	-1	1
9	13	-4	4
16	19	-3	3
9	11	-2	2
11	15	-4	4
90	90	0	0

Average	-0.85	1.85
Standard Deviation	2.17	1.42
n	20	20
df	19	19
α	0.05	0.05
t	2.093	2.093
tails	2	2
min	1.96	1.19
max	+0.06	2.51
error range	±1.01	±0.66

OK Known oil content estimated at ±1% oil
OD Determined oil content
* Data points excluded

TABLE E-2. STATISTICAL SUMMARY OF STRATIFIED SAMPLE TEST RESULTS USING METHOD 1

OK % Oil	OD % Oil	OK-OD % Oil	|OK-OD| % Oil
74	73	1	1
64	60	4	4
54	60	-6	6
67	66	1	1
46	46	0	0
56	54	2	2
77	71	6	6
87	83	4	4
66	65	1	1
82	79	3	3
40	40	0	0
50	48	2	2
44	45	-1	1
37	35	2	2
37	39	0	0
31	29	2	2
23	21	2	2
13	9	4	4
19	18	1	1
11	10	1	1
15	10	5	5
90	91	-1	1

Average	1.50	2.23
Standard Deviation	2.45	1.81
n	22	22
df	21	21
α	0.05	0.05
t	2.080	2.080
min	0.42	1.43
max	2.58	3.03
error range	±1.08	±0.80

OK known oil content estimated at ±1% oil
OD determined oil content

TABLE B-3. STATISTICAL SUMMARY OF STRATIFIED SAMPLE TEST RESULTS USING METHOD 2

OK % Oil	OD % Oil	OK-OD % Oil	| OK-OD % Oil |
74	75	-1	1
64	72	-8	8
54	50	4	4
67	68	-1	1
46	68	-22	22
56	68	-12	12
77	75	2	2
87	83	4	4
66	63	3	3
82	79	3	3
40	34	6	6
50	55	-5	5
44	50	-6	6
37	40	-3	3
39	40	-1	1
31	34	-3	3
23	20	3	3
13	12	1	1
19	20	-1	1
11	12	-1	1
15	8	7	7
90	91	-1	1

Average	-1.45	4.45
Standard Deviation	6.32	4.72
n	22	22
df	21	21
α	0.05	0.05
t	2.080	2.080
min	-4.26	2.36
max	1.35	6.55
error range	±2.80	±2.09

OK known oil content estimated at ±1% oil
OD determined oil content

TABLE B-4. STATISTICAL SUMMARY OF STRATIFIED SAMPLE TEST RESULTS USING METHOD 3

OK % Oil	OD % Oil	OK-OD % Oil	|OK-OD| % Oil
74	87	-13	13
64	82	-18	18
54	77	-23	23
67	82	-15	15
46	54	- 8	8
56	68	-12	12
77	99	-22	22
87	99	-12	12
66	83	-17	17
82	97	-15	15
40	51	-11	11
50	74	-24	24
44	56	-12	12
37	50	-13	13
39	52	-13	13
31	40	- 9	11
23	33	-10	10
13	20	- 7	7
19	25	- 6	6
11	17	- 6	6
15	19	- 4	4
90	100	-10	10
	Average	-12.73	12.73
	Standard Deviation	5.35	5.35
	n	22	22
	df	21	21
	α	0.05	0.05
	t	2.080	2.080
	min	-15.10	10.35
	max	-10.35	15.10
	error range	±2.37	±2.37

OK known oil content estimated at ±1% oil.
OD determined oil content

TABLE B-5. STATISTICAL SUMMARY OF STRATIFIED SAMPLE TEST RESULTS USING METHOD 4

OK % Oil	OD % Oil	OK-OD % Oil	OK-OD % Oil
74	74	0	0
64	70	-6	6
66	60	6	6
67	68	-1	1
46	59	-13	13
56	58	-2	2
77	79	-2	2
87	86	1	1
66	63	3	3
82	81	1	1
40	41	-1	1
50	45	5	5
44	48	-4	4
37	32	5	5
39	45	-6	6
31	30	1	1
23	19	4	4
13	6	7	7
19	11	8	8
11	4	7	7
15	6	9	9
90	98	-8	8
	Average	0.64	4.55
	Standard Deviation	5.56	3.26
	n	22	22
	df	21	21
	α	0.05	0.05
	t	2.080	2.080
	min	-1.82	3.10
	max	3.10	5.99
	error range	±2.46	±1.44

OK known oil content estimated at ±1% oil
OD determined oil content

TABLE B-6. STATISTICAL SUMMARY OF PITOT-TUBE SAMPLING PORT

OK % Oil	OD % Oil	OK-OD % Oil	|OK-OD % Oil|
56	56	4	4
60	58	2	2
97	97	0	0
86	84	2	2
78	71	7	7
75	68	7	7
41	40	1	1
36	42	-6	6
29	31	-2	2
28	32	-4	4
21	21	0	0
22	28	-6	6
	Average	0.42	3.42
	Standard Deviation	4.21	2.50
	n	12	12
	α	0.05	0.05
	t	2.201	2.201
	min	-2.26	1.83
	max	3.10	5.01
	error range	±2.46	±1.59

OK known oil content estimated at + ±1% oil
OD determined oil content

TABLE B-7. STATISTICAL SUMMARY OF SLOTTED TUBE SAMPLING PORT

OK % Oil	OD % Oil	OK-OD % Oil	|OK-OD| % Oil
56	52	4	4
60	59	1	1
97	97	0	0
86	84	2	2
78	73	5	5
75	73	2	2
41	46	-5	5
36	41	-5	5
29	36	-7	7
28	31	-3	3
21	21	0	0
22	25	-3	3
	Average	-0.75	3.08
	Standard Deviation	3.63	2.10
	α	0.05	0.05
	t	2.201	2.201
	min	-3.07	1.75
	max	1.57	4.42
	error range	2.32	1.33

OK known oil content estimated at + ±1% oil
OD determined oil content