

Final report for “Modular Oil Absorbent Bilge Pump”
Sea Grant Project No. FRGP 2002-29

Report prepared by Brian P. Lilliston, June 30, 2003



*An investment in Virginia's
natural resources, funded by the
Virginia Sea-grant Program*



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Summary

The intent of this project was to create a modular housing that could be used with standard bilge pumps to reduce oily discharges in Virginia waterways. In designing the housing the most important factor would be the total percentage of petroleum reduction that would be realized. However, in order to reap the benefits of the housings they would have to meet certain requirements in order for boat owners to install them in a significant number of vessels.

These end-user requirements include:

- Cost
- Life expectancy
- Ease of use and maintenance
- Performance in contaminant removal
- Minimal interference with normal water removal by bilge pumps

The selected design was a compromise of several configurations originally drawn for the project. This design was chosen because it cannot restrict the pump flow in an emergency condition. In conversations with vessel captains prior to submitting the original proposal, it was clear that any potential for pump restriction would make the housing assembly “worthless” to boat owners. The current housing that was completed and tested filters water until levels exceed three inches. If the vessel is taking on water faster than it can be filtered and discharged, the pump will allow water to by-pass the filter media and pump at 100% flow rate.

Our design goals have been achieved with an assembly that is compact and has a life expectancy of 10 years in the bilge of any vessel. The housing will accept any bilge pump up to 2500 gph and only requires a nut driver to install the housing or for replacement of the pump, automatic switch, or filter element.

Our efficiency goals have been surpassed ten fold. The EPA approved water tests of contaminant removal were carried out by Oakwood Scientific Laboratory, located in Mechanicsville, Virginia. These tests show a contaminant removal rate of approximately 80% by volume of petroleum products. This reduction multiplied by hundreds of boats would be a substantial aide in protecting our marine habitat and therefore our fishery resources.

Design and Construction

The physical design of the pump housing began with material selection. Regardless of the purpose or finished size of the unit, the materials chosen had to endure a ten year lifespan and provide ease of construction, repair, and modification. Secondary considerations must include price and difficulty of manufacture.

Aluminum has the characteristics outlined above and was used predominantly in the unit. Exceptions include the screen, hinge for top lid, and miscellaneous screws. The screen used is made of silicon bronze and is an assembly used for screening raw water intakes and is readily available at boating supply stores. The stainless steel piano hinge and hardware are also standard items readily available.

The housing was made to accept 1500-2000 gph pumps from a variety of manufacturers with a Rule brand pump being used. The housing was kept as small as possible as not to hinder installation in smaller boats with limited bilge access.

For ease of maintenance the lid was attached with a hinge. For specific applications a removable top would also be an option.

Unassembled pump unit consisting of standard rule 1500 bilge pump, silicon bronze commercial intake screens, and aluminum lid and main housing.



Testing of Pump Unit

The housing is screened in the rear panel, two lid panels, and lower front panel, but by design filter material is only packed in the lower 3” of the front and rear. In normal conditions oil drips will slowly accumulate and therefore will have adequate time to be absorbed by the filter. If water comes into the bilge faster than it can be cycled out, the water will by-pass the filter portion of the system after a three inch water depth. It was important to maintain screening above the three inch water depth as a way to improve the reliability of the pump. The screening will prevent any trash or debris from obstructing the pump or switch action.

There were a variety of conditions that the pump could have been tested under. Although testing a flood condition would give the filter the least amount of time to absorb oil ... this worst case scenario was chosen for testing over a slow accumulation of water and oil such as a hull leak or packing leak.

Two aluminum tanks were constructed measuring 14” x 36” x 48” which hold 100 gallons each. One quart of 15w-40 motor oil was added into one of the empty tanks. This tank was then filled to the 100 gallon mark with tap water. As soon as the tank was full the pump unit was placed into the tank and pumping began.



Test Results

The pump took four minutes and thirty-five seconds to clear the contaminated tank down to the filtering level of 3". This flow rate is equal to the flow rate of the same pump, in the same tank, using the same power source and discharge hose. The filtering and removal of the remaining water (approximately 8.7 gallons) took twenty-one minutes to be pumped into the containment tank.

When in filtering mode, the water will permeate the filter material and fill the aluminum housing until the automatic switch turns on the pump. The bilge pump discharges a short pulse of water every 4-5 seconds and the cycle repeats until the tank is empty.

As soon as the pumping was completed, a water sample was taken by putting the sample bottle at the surface of the containment tank and submerging the opening half way into the surface of the water. This first sample was tested and contained 450 milligrams of oil per liter (450 parts per million of oil)

Five minutes after pumping a second sample was taken using the same method. This sample was tested and contained 122 milligrams of oil per liter (122 parts per million of oil).

Thirty minutes after pumping a third sample was taken using the same method. This sample was tested and contained 2.3 milligrams of oil per liter (2.3 parts per million of oil).



Overall Effectiveness of Unit

With each test bottle filled, contamination levels were decreasing as one would expect levels of contamination to increase due to more oil floating to the surface. This suggests that the measured quantities in the samples made up the bulk of oil pumped into the containment tank, which in a boating application would represent the Chesapeake Bay.

The 32 ounces of oil added before the test was equal to **907, 184.74 milligrams**.

Oil was removed as follows

Sample 1	0 minutes after pumping	450.00 milligrams/liter
Sample 2	5 minutes after pumping	122.00 milligrams/liter
Sample 3	30 minutes after pumping	2.30 milligrams/liter

Putting our removal results into a percentage has been troubling due to the fact that the entire 100 gallon / 378 liter sample could not be tested to give an exact percentage of remaining oil. If a percentage is required, I would recommend playing “devils advocate” and multiplying the highest sample of 450 milligrams/liter by the entire containment tank volume of 100 gallons or 378 liters.

450 mg/liter x 378 liter =	170,100.00 milligrams
Total of oil introduced	907,184.74 milligrams

With these “worst case” statistics it would mean that only 18.5% of the oil remained in the discharge.
Efficiency rate of removal 81.5%

NOTE

In planning the testing we had only anticipated reducing pollutants by absorbing oil into the filter material. After conducting our tests we discovered that although the filter could not absorb oil at a rapid rate, it did however block the oil from entering the pump and therefore being pumped overboard. With this added benefit, oil that was not absorbed was not necessarily pumped out, but rather left in the bilge where the boat owner would be given a chance to clean and remove it.



Recommendations

1. With a limited amount of tooling the housing assembly could be fabricated and sold to boat owners for approximately one-hundred dollars. More filters installed in boats would greatly reduce discharges into our waters.
2. The most annoying aspect of the housing to the boat owners that have used in in trials for the past year, is the time it takes to replace the filter material. A quick change cartridge would make the entire unit more appealing to boat owners.
3. Jack Ward of Jacks Marine Repair, Karl Wendley of Deep Creek Marine Service, and Red Godsey of Godseys Marine suggested that most people would be reluctant to install the housing out of concern for the environment. They all also noted that a more convenient filter change would make the housing more appealing. Karl Wendley suggested testing the unit with a 1” hole bored through the bottom of the housing to allow the lower level water to enter the pump.
4. Willie Smullin owner of F/V “Desparado” and F/V “Blown Away” had the housings installed on each of his commercial fishing boats measuring 44’ and 38’ respectively. The housings performed well and of special interest the filters absorbed bilge sheen that appeared on windy days due to water movement in the bilge. Again a “quick change” filter element was desired by Mr. Smullin and his captain.

OAKWOOD SCIENTIFIC LABORATORY
State Certified Bacteriological Water Testing



7102 Pole Green Road
Mechanicsville, VA 23116

(804) 730-3263
1-800-582-5211

Date: 7/21/03

Chemical Examination of Water

Sample Number: 071203-7 Lilliston

Requested by: Brian Lilliston (sample 02 filtered Omin. after)

Sample Origin: Mr. Brian Lilliston
183216 Plantation Rd.
Onancock, VA 23417

Results: 450 mg oil & grease per liter of water (450 ppm oil & grease in water)

Partition-Gravimetric Method 5520 B. Standard Methods APHA 19th ed.
ppm = parts per million, mg/l=milligrams per liter, "<" symbol is read
less than or undetectable at that level.

In the determination of **oil and grease**, an absolute quantity of a specific substance is not measured. Rather, groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in an oil and grease solvent. It is important that this limitation be understood clearly. This group includes other material extracted by the solvent from an acidified sample (such as sulfur compounds, certain organic dyes, and chlorophyll) and not volatilized during the test. Oils and greases are thus defined by the method used for determination. Extraction of #2 heating oil by freon 113 is about 93% efficient so actual oil concentration is slightly higher. This method is useful for detecting leaking oil tanks and spills into drinking water wells, aquifer contamination and waste water treatment systems.

The above services were performed and the report prepared in accordance with accepted laboratory practices, and makes no other warranties, either expressed or implied, as to the professional advice provided herein.

Respectfully,

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Dr. Ronald R. Weik, Ph.D.
Director, Microbiology

A handwritten signature in black ink, appearing to read "Simonetta M. Weik".

Simonetta M. Weik
Assistant Director

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State Certified Bacteriological Water Testing



7102 Pole Green Road
Mechanicsville, VA 23116

(804) 730-3263
1-800-582-5211

Date: 7/21/03

Chemical Examination of Water

Sample Number: 071203-8 Lilliston

Requested by: Brian Lilliston (sample 03 filtered 5 min after)

Sample Origin: Mr. Brian Lilliston
183216 Plantation Rd.
Onancock, VA 23417

Results: 122 mg oil & grease per liter of water (122 ppm oil & grease in water)

Partition-Gravimetric Method 5520 B. Standard Methods APHA 19th ed.
ppm = parts per million, mg/l=milligrams per liter, "<" symbol is read
less than or undetectable at that level.

In the determination of **oil and grease**, an absolute quantity of a specific substance is not measured. Rather, groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in an oil and grease solvent. It is important that this limitation be understood clearly. This group includes other material extracted by the solvent from an acidified sample (such as sulfur compounds, certain organic dyes, and chlorophyll) and not volatilized during the test. Oils and greases are thus defined by the method used for determination. Extraction of #2 heating oil by freon 113 is about 93% efficient so actual oil concentration is slightly higher. This method is useful for detecting leaking oil tanks and spills into drinking water wells, aquifer contamination and waste water treatment systems.

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State Certified Bacteriological Water Testing



7102 Pole Green Road
Mechanicsville, VA 23116

(804) 730-3263
1-800-582-5211

Date: 7/21/03

Chemical Examination of Water

Sample Number: 071203-9 Lilliston

Requested by: Brian Lilliston (sample 04 filtered 30 min after)

Sample Origin: Mr. Brian Lilliston
183216 Plantation Rd.
Onancock, VA 23417

Results: 2.3 mg oil & grease per liter of water (2.3 ppm oil & grease in water)

Partition-Gravimetric Method 5520 B. Standard Methods APHA 19th ed.
ppm = parts per million, mg/l=milligrams per liter, "<" symbol is read
less than or undetectable at that level.

In the determination of **oil and grease**, an absolute quantity of a specific substance is not measured. Rather, groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in an oil and grease solvent. It is important that this limitation be understood clearly. This group includes other material extracted by the solvent from an acidified sample (such as sulfur compounds, certain organic dyes, and chlorophyll) and not volatilized during the test. Oils and greases are thus defined by the method used for determination. Extraction of #2 heating oil by freon 113 is about 93% efficient so actual oil concentration is slightly higher. This method is useful for detecting leaking oil tanks and spills into drinking water wells, aquifer contamination and waste water treatment systems.

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Director, Microbiology

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Simonetta M. Weik
Assistant Director

Reagents

In addition to reagents *a*, *c*, *e*, *g*, and *i* of Section 5510B:

- a. XAD resin,† approximately 250- μ m size.
- b. Hexane.
- c. Methanol.
- d. Acetonitrile.

Procedure

- a. *Sample collection and preservation*: See Section 5510B.4a.
- b. *Preparation of XAD resin*: Clean resin by successive washings with 0.1N NaOH for 5 d. Extract resin sequentially in a Soxhlet extractor with hexane, methanol, acetonitrile, and methanol, for 24 h each. Pack clean resin into a 0.2- \times 25-cm glass column that has a 2-mm length of glass wool in one end. After filling, cap column with another 2-mm length of glass wool. Wet dry column with methanol. When the air has been displaced, pump distilled water through the column until the effluent concentration of DOC decreases to 0.5 mg/L (approximately 20 bed volumes).
- c. *Chromatography*: Preclean column with three cycles of 0.1N NaOH and 0.1N HCl just before pumping sample into column. Pre-equilibrate column saturated with 0.1N HCl. Acidify sample to pH 2 with concentrated HCl, and pump it onto the column at rate 1.0 mL/min. Save column effluent for DOC analysis. Significant concentrations of DOC in the effluent can indicate that the column was overloaded and that a smaller sample volume should be used. Colored organic acids adsorb to the top of the column. Back-elute (reverse flow) the column with 0.1N NaOH at 0.2

mL/min and collect eluate in a graduated, conical test tube until it becomes colorless (about 2 mL). Acidify with conc H_3PO_4 to a pH of 2 or less (about 2 to 3 drops) and remove dissolved carbon dioxide (inorganic carbon) by purging with nitrogen for 10 min. Avoid exposure of alkaline samples to air (i.e. acidify immediately) to minimize contamination with CO_2 . Determine volume and DOC of acidified column effluent.

After eluting and collecting AHS from the column with back-elution using 0.1N NaOH, continue rinsing with about 20 bed volumes of the basic solution. Rinse with water for about 20 bed volumes. Repeat the triplicate acid/base column precleaning procedure described above, then reuse the column to analyze a replicate sample. Process two portions of water by the same procedure to serve as controls.

The XAD column may be reused to analyze subsequent samples and controls if the triplicate acid/base precleaning procedure is repeated immediately before analysis of each replicate. Replace the column if recovery is poor or the resin becomes discolored.

5. Calculation

Calculate the concentration of AHS as given in 5510B.5.

6. Precision and Bias

For seven single-operator analyses, the relative standard deviation of triplicate samples (about 10 mg/L as AHS) ranged from 0.9 to 20.7% with an average of 5.4% ($n = 7$).

For seven single-operator analyses, recoveries ranged from 15.1 to 71.0% with an average of 51.6% and a relative standard deviation of 35.1%.

† AD-7, or equivalent.

5520 OIL AND GREASE*

5520 A. Introduction

In the determination of oil and grease, an absolute quantity of a specific substance is not measured. Rather, groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in an organic extracting solvent. "Oil and grease" is defined as any material recovered as a substance soluble in the solvent. It includes other material extracted by the solvent from an acidified sample (such as sulfur compounds, certain organic dyes, and chlorophyll) and that volatilized during the test. The 12th edition of *Standard Methods* prescribed the use of petroleum ether as the solvent for natural and treated waters and *n*-hexane for polluted waters. The 13th edition added trichlorotrifluoroethane as an optional solvent for all sample types. In the 14th through the 17th editions, only trichlorotrifluoroethane was specified. Trichlorotrifluoroethane until the prescribed solvent; however, because of environmental problems associated with chlorofluorocarbons, an alternative solvent (80% *n*-hexane and 20% methyl-*tert*-butyl ether) is included in gravimetric methods. This alternative solvent mixture produces results very similar to those obtained on the same samples

with trichlorotrifluoroethane.† Solvent-recovery techniques have been included and solvent recycling is strongly recommended.

It is important to understand that, unlike some constituents that represent distinct chemical elements, ions, compounds, or groups of compounds, oils and greases are defined by the method used for their determination.

The methods presented here are suitable for biological lipids and mineral hydrocarbons. They also may be suitable for most industrial wastewaters or treated effluents containing these materials, although sample complexity may result in either low or high results because of lack of analytical specificity. The method is not applicable to measurement of low-boiling fractions that volatilize at temperatures below 70°C when trichlorotrifluoroethane is used or below 85°C when the *n*-hexane/methyl-*tert*-butyl ether solvent mix is used.

1. Significance

Certain constituents measured by the oil and grease analysis may influence wastewater treatment systems. If present in ex-

* Approved by Standard Methods Committee, 1991.

† U.S. EPA currently is proposing the use of *n*-hexane only in lieu of the hexane-MtBE method.

Standard Methods 19th ed. 1995

cessive amounts, they may interfere with aerobic and anaerobic biological processes and lead to decreased wastewater treatment efficiency. When discharged in wastewater or treated effluents, they may cause surface films and shoreline deposits leading to environmental degradation.

A knowledge of the quantity of oil and grease present is helpful in proper design and operation of wastewater treatment systems and also may call attention to certain treatment difficulties.

In the absence of specially modified industrial products, oil and grease is composed primarily of fatty matter from animal and vegetable sources and from hydrocarbons of petroleum origin. The portion of oil and grease from each of these two major sources can be determined with Method 5520F. A knowledge of the relative composition of a sample minimizes the difficulty in determining the major source of the material and simplifies the correction of oil and grease problems in wastewater treatment plant operation and stream pollution abatement.

2. Selection of Method

For liquid samples, three methods are presented: the partition-gravimetric method (B), the partition-infrared method (C), and the Soxhlet method (D). Method C is designed for samples that might contain volatile hydrocarbons that otherwise would be lost in the solvent-removal operations of the gravimetric procedure. Method D is the method of choice when relatively polar, heavy petroleum fractions are present, or when the levels of nonvolatile greases may challenge the solubility limit of the solvent. For low levels of oil and grease (< 10 mg/L), Method C is the method of choice because gravimetric methods do not provide the needed precision.

Method E is a modification of the Soxhlet method and is suitable for sludges and similar materials. Method F can be used in conjunction with Methods B, C, D, or E to obtain a hydrocarbon measurement in addition to, or instead of, the oil and grease measurement. This method separates hydrocarbons from the total oil and grease on the basis of polarity.

3. Sample Collection, Preservation, and Storage

Collect a representative sample in a wide-mouth glass bottle that has been washed with soap, rinsed with water, and finally rinsed with solvent to remove any residues that might interfere with the analysis. Collect a separate sample for an oil and grease determination. Do not overfill the sample container, and do not subdivide the sample in the laboratory. If analysis is to be delayed or more than 2 h, acidify to pH 2 or lower with 1:1 HCl and refrigerate. When information is required about average grease concentration over an extended period, examine individual portions collected at prescribed time intervals to eliminate losses of grease on sampling equipment during collection of a composite sample.

In sampling sludges, take every possible precaution to obtain a representative sample. When analysis cannot be made within

2 h, preserve samples with 1 mL conc HCl/80 g sample and refrigerate. Never preserve samples with CHCl₃ or sodium benzoate.

4. Interferences

a. Organic solvents have the ability to dissolve not only oil and grease but also other organic substances. Any filterable solvent-soluble substances (e.g., elemental sulfur, complex aromatic compounds, hydrocarbon derivatives of chlorine, sulfur, and nitrogen, and certain organic dyes) that are extracted and recovered are defined as oil and grease. No known solvent will dissolve selectively only oil and grease. Heavier residuals of petroleum may contain a significant portion of materials that are not solvent-extractable. The method is entirely empirical; duplicate results with a high degree of precision can be obtained only by strict adherence to all details.

b. For Methods 5520B, D, E, and F, solvent removal results in the loss of short-chain hydrocarbons and simple aromatics by volatilization. Significant portions of petroleum distillates from gasoline through No. 2 fuel oil are lost in this process. Adhere strictly to sample drying time, to standardize gradual loss of weight due to volatilization. For Methods 5520B, D, E, and F, during the cooling of the distillation flask and extracted material, a gradual increase in weight may be observed, presumably due to the absorption of water if a desiccator is not used. For Method 5520C use of an infrared detector offers a degree of selectivity to overcome some coextracted interferences (§ 4a). For Methods 5520D and E, use exactly the specified rate and time of extraction in the Soxhlet apparatus because of varying solubilities of different greases. For Method 5520F, the more polar hydrocarbons, such as complex aromatic compounds and hydrocarbon derivatives of chlorine, sulfur, and nitrogen, may be adsorbed by the silica gel. Extracted compounds other than hydrocarbons and fatty matter also interfere.

c. Alternative techniques may be needed for some samples if intractable emulsions form that cannot be broken by centrifugation. Such samples may include effluents from pulp/paper processing and zeolite manufacturing. Determine such modifications on a case-by-case basis.

d. Some sample matrices can increase the amount of water partitioned into the organic extraction fluid. When the extraction solvent from this type of sample is dried with sodium sulfate, the drying capacity of the sodium sulfate can be exceeded, thus allowing sodium sulfate to dissolve and pass into the tared flask. After drying, sodium sulfate crystals will be visible in the flask. The sodium sulfate that passes into the flask becomes a positive interference in methods employing gravimetric measurement. If crystals are observed in the tared flask after drying, redissolve any oil and grease with 30 mL of extraction solvent, then drain the solvent through a funnel containing a solvent-rinsed filter paper into a clean, tared flask. Rinse the first flask twice more, combining all solvent in the new flask, and treat as an extracted sample.

5520 B. Partition-Gravimetric Method

General Discussion

Dissolved or emulsified oil and grease is extracted from water by intimate contact with an extracting solvent. Some extracta-

bles, especially unsaturated fats and fatty acids, oxidize readily; hence, special precautions regarding temperature and solvent vapor displacement are included to minimize this effect. Organic solvents shaken with some samples may form an emulsion that

s very difficult to break. This method includes a means for handling such emulsions. Recovery of trichlorotrifluoroethane and other solvents is discussed; recovery can reduce both vapor emissions to the atmosphere and costs of solvent supply.

2. Apparatus

- Separatory funnel, 2-L, with TFE* stopcock.
- Distilling flask, 125-mL.
- Liquid funnel, glass.
- Filter paper, 11-cm diam.†
- Centrifuge, capable of spinning at least four 100-mL glass centrifuge tubes at 2400 rpm or more.
- Centrifuge tubes, 100-mL, glass.
- Water bath, capable of maintaining 85°C.
- Vacuum pump or other source of vacuum.
- Distilling adapter with drip tip. Setup of distillate recovery apparatus is shown in Figure 5520:1. Alternatively, use commercially available solvent recovery equipment.
- Ice bath.
- Waste receptacle, for used solvent.
- Desiccator.

3. Reagents

- Hydrochloric acid, HCl, 1 + 1.
- Trichlorotrifluoroethane (1,1,2-trichloro-1,2,2-trifluoroethane), boiling point 47°C. The solvent should leave no measurable residue on evaporation; distill if necessary. Do not use any plastic tubing to transfer solvent between containers.
- n*-Hexane, boiling point 69°C. The solvent should leave no measurable residue on evaporation; distill if necessary. Do not use any plastic tubing to transfer solvent between containers.
- Methyl-*tert*-butyl ether, boiling point 55°C to 56°C. The solvent should leave no measurable residue on evaporation; distill if necessary. Do not use any plastic tubing to transfer solvent between containers.
- Sodium sulfate, Na₂SO₄, anhydrous crystal.

4. Procedure

When a sample is brought into the laboratory, mark sample level on bottle for later determination of sample volume. If sample has not been acidified previously (see Section 5520A.3), acidify with 1:1 HCl to pH 2 or lower (generally, 5 mL is sufficient). Transfer sample to a separatory funnel. Carefully rinse sample bottle with 30 mL of extracting solvent and add solvent washings to separatory funnel. Shake vigorously for 2 min. Let layers separate. Next, depending on the solvent used (*n*-hexane/methyl-*tert*-butyl ether will be upper layer or trichlorotrifluoroethane will be lower layer) drain solvent layer through a funnel containing a filter paper and 10 g Na₂SO₄, both of which have been solvent-rinsed, into a clean, tared distilling flask. If a clear solvent layer cannot be obtained and an emulsion of more than about 5 mL exists, drain emulsion and solvent layers into a glass centrifuge tube and centrifuge for 5 min at approximately 2400 rpm. Transfer centrifuged material to an appropriate separatory funnel and drain solvent layer through a funnel with a filter paper

* Teflon or equivalent.

† Whatman No. 40 or equivalent.

and 10 g Na₂SO₄, both of which have been prerinced, into a clean, tared distilling flask. Recombine aqueous layers and any remaining emulsion or solids in separatory funnel. For samples with <5 mL of emulsion, drain only the clear solvent through a funnel with pre-moistened filter paper and 10 g Na₂SO₄. Recombine aqueous layers and any remaining emulsion or solids in separatory funnel. Extract twice more with 30 mL solvent each time, but first rinse sample container with each solvent portion. Repeat centrifugation step if emulsion persists in subsequent extraction steps. Combine extracts in tared distilling flask, and include in flask a final rinsing of filter and Na₂SO₄ with an additional 10 to 20 mL solvent. Distill solvent from flask in a water bath at 70°C for trichlorotrifluoroethane or 85°C for the *n*-hexane/methyl-*tert*-butyl ether mixture. To maximize solvent recovery, fit distillation flask with a distillation adapter equipped with a drip tip and collect solvent in an ice-bath-cooled receiver (Figure 5520:1). When visible solvent condensation stops, remove flask from water bath. Cover water bath and dry flasks on top of cover at 70°C for trichlorotrifluoroethane or 85°C for the *n*-hexane/methyl-*tert*-butyl ether mixture for 15 min. Draw air through flask with an applied vacuum for 15 min. Cool in desiccator for at least 30 min and weigh. To determine initial sample volume, fill sample bottle to mark with water and then pour water into a 1-L graduated cylinder.

5. Calculation

If the organic solvent is free of residue, the gain in weight of the tared distilling flask is due mainly to oil and grease. Total gain in weight, *A*, of tared flask less calculated residue, *B*, from solvent blank is the amount of oil and grease in the sample:

$$\text{mg oil and grease/L} = \frac{(A - B) \times 1000}{\text{mL sample}}$$

6. Precision and Bias

Methods B, C, and D, with trichlorotrifluoroethane as solvent, were used by a single laboratory to test a sewage sample. By this

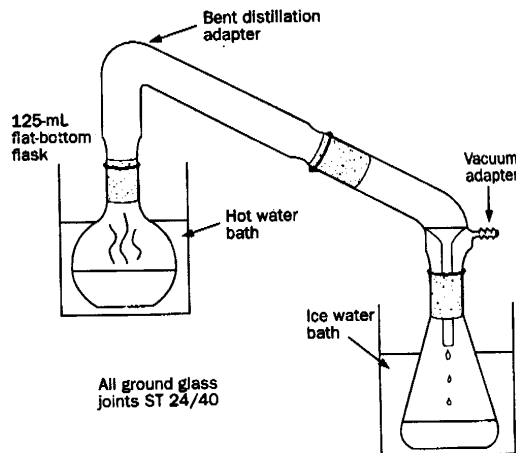


Figure 5520:1. Distillate recovery apparatus.

method the oil and grease concentration was 12.6 mg/L. When 1-L portions of the sewage were dosed with 14.0 mg of a mixture of No. 2 fuel oil and Wesson oil, recovery of added oils was 93% with a standard deviation of 0.9 mg.

Method B was tested by a single laboratory on a raw sewage sample using both extraction solvents. By this method the oil and grease concentration was 20.8 mg/L with trichlorotrifluoroethane and 22.4 mg/L with the 80:20 hexane/methyl-*tert*-butyl ether mixture. When samples were dosed with 30 mg Fisher

Heavy Mineral Oil, recovery of added oil was 78.9% with a standard deviation of 0.8 mg/L for trichlorotrifluoroethane and 84.2% with a standard deviation of 1.2 mg/L for hexane/methyl-*tert*-butyl ether.

7. Bibliography

KIRSCHMAN, H.D. & R. POMEROY. 1949. Determination of oil in oil field waste waters. *Anal. Chem.* 21:793.

5520 C. Partition-Infrared Method

1. General Discussion

a. Principle: The use of trichlorotrifluoroethane as extraction solvent allows absorbance of the carbon-hydrogen bond in the infrared to be used to measure oil and grease. The sample extraction procedure for this method is identical to that when trichlorotrifluoroethane is used in Method B. Elimination of the evaporation step permits infrared detection of many relatively volatile hydrocarbons. Thus, the lighter petroleum distillates, with the exception of gasoline, may be measured accurately. With adequate instrumentation, as little as 0.2 mg oil and grease/L can be measured.

b. Definitions: A "known oil" is defined as a sample of oil and/or grease that represents the only material of that type used or manufactured in the processes represented by a wastewater. An "unknown oil" is defined as one for which a representative sample of the oil or grease is not available for preparation of a standard.

2. Apparatus

- a. Separatory funnel*, 2-L, with TFE* stopcock.
- b. Volumetric flask*, 100-mL.
- c. Liquid funnel*, glass.
- d. Filter paper*, 11-cm diam.†
- e. Centrifuge*, capable of spinning at least four 100-mL glass centrifuge tubes at 2400 rpm or more.
- f. Centrifuge tubes*, 100-mL, glass.
- g. Infrared spectrophotometer*, double-beam, recording.
- h. Cells*, near-infrared silica.

3. Reagents

- a. Hydrochloric acid*, HCl, 1 + 1.
- b. Trichlorotrifluoroethane:* See Section 5520B.3b.
- c. Sodium sulfate*, Na₂SO₄, anhydrous, crystal.
- d. Reference oil:* Prepare a mixture, by volume, of 37.5% iso-octane, 37.5% hexadecane, and 25.0% benzene. Store in sealed container to prevent evaporation.

4. Procedure

Refer to Section 5520B.4 for sample handling and extraction and for method of dealing with sample emulsions. Collect com-

* Teflon or equivalent.

† Whatman No. 40 or equivalent.

posed extracts in a 100-mL volumetric flask and adjust final volume to 100 mL with solvent.

Prepare a stock solution of known oil by rapidly transferring about 1 mL (0.5 to 1.0 g) of the oil or grease to a tared 100-mL volumetric flask. Stopper flask and weigh to nearest milligram. Add solvent to dissolve and dilute to mark. If the oil identity is unknown (5520C.1b) use the reference oil (5520C.3d) as the standard. Using volumetric techniques, prepare a series of standards over the range of interest. Select a pair of matched near-infrared silica cells. A 1-cm-path-length cell is appropriate for a working range of about 4 to 40 mg. Scan standards and samples from 3200 cm⁻¹ to 2700 cm⁻¹ with solvent in the reference beam and record results on absorbance paper. Measure absorbances of samples and standards by constructing a straight base line over the scan range and measuring absorbance of the peak maximum at 2930 cm⁻¹ and subtracting baseline absorbance at that point. If the absorbance exceeds 0.8 for a sample, select a shorter path length or dilute as required. Use scans of standards to prepare a calibration curve.

5. Calculation

$$\text{mg oil and grease/L} = \frac{A \times 1000}{\text{mL sample}}$$

where:

A = mg of oil or grease in extract as determined from calibration curve.

6. Precision and Bias

Methods B, C, and D, with trichlorotrifluoroethane as solvent, were used by a single laboratory to test a sewage sample. By this method the oil and grease concentration was 17.5 mg/L. When 1-L portions of the sewage were dosed with 14.0 mg of a mixture of No. 2 fuel oil and Wesson oil, the recovery of added oils was 99% with a standard deviation of 1.4 mg.

7. Bibliography

GRUENFELD, M. 1973. Extraction of dispersed oils from water for quantitative analysis by infrared spectrophotometry. *Environ. Sci. Technol.* 7:636.

Test Results

The pump took four minutes and thirty-five seconds to clear the contaminated tank down to the filtering level of 3". This flow rate is equal to the flow rate of the same pump, in the same tank, using the same power source and discharge hose. The filtering and removal of the remaining water (approximately 8.7 gallons) took twenty-one minutes to be pumped into the containment tank.

When in filtering mode, the water will permeate the filter material and fill the aluminum housing until the automatic switch turns on the pump. The bilge pump discharges a short pulse of water every 4-5 seconds and the cycle repeats until the tank is empty.

As soon as the pumping was completed, a water sample was taken by putting the sample bottle at the surface of the containment tank and submerging the opening half way into the surface of the water. This first sample was tested and contained 450 milligrams of oil per liter (450 parts per million of oil)

Five minutes after pumping a second sample was taken using the same method. This sample was tested and contained 122 milligrams of oil per liter (122 parts per million of oil).

Thirty minutes after pumping a third sample was taken using the same method. This sample was tested and contained 2.3 milligrams of oil per liter (2.3 parts per million of oil).



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