

## ENGINE FUEL STORAGE OPERATING METHODS

CONTENTS	PAGE	CONTENTS	PAGE
1. GENERAL . . . . .	1	Figures	
2. APPARATUS . . . . .	2	1. Typical Sample Container Information Label . . . . .	6
3. FUEL REQUIREMENTS . . . . .	2		
A. Diesel and Gas Turbine Engines . . . . .	2	1. GENERAL	
B. Gasoline Engines . . . . .	3	1.01 This section lists the requirements for engine fuels for use in stationary engines, describes the use of an inhibitor to stabilize fuels, recommends fuel storage system operating practices, covers methods of detecting and removing water and solids (both organic and inorganic) in fuel tanks, and describes test procedures and methods.	
4. RECOMMENDED FUEL STORAGE PRACTICES . . . . .	3	1.02 Revision arrows are used to emphasize significant changes. The Equipment Test List is affected. The reason for reissue are listed below:	
A. Maintenance Considerations . . . . .	3	(a) To revise the requirements of engine fuels and fuel storage to clarify the occasions when chemical analysis is preferred over filtration.	
B. Laboratory Test Methods . . . . .	3	(b) To clarify and revise the methods for water detection and removal, fuel sampling, fuel filtration, and use of fuel inhibitor.	
5. WATER DETECTION AND REMOVAL . . . . .	3	(c) To revise the recommended test methods and procedures to be used by the testing laboratory.	
6. FUEL SAMPLING . . . . .	4	(d) To remove Part 10.	
7. FUEL INHIBITOR . . . . .	5	1.03 Virtually all gas turbine or diesel engine fuels are unsatisfactory for long term storage as delivered by the fuel supplier. KS-21923, L1, fuel inhibitor must generally be added to retard sludge formation and prevent internal corrosion of metal fuel storage tanks. The inhibitor contains a <i>stabilizer</i> to retard sludge formation; a <i>dispersant</i> to keep any moderate amount of sludge that does form from ag-	
8. ON-SITE FUEL FILTRATION . . . . .	5		
9. DESCRIPTION OF FUEL TEST PROCEDURES AND LABORATORY METHODS . . . . .	7		
A. Accelerated Aging of Petroleum Distillate Fuel . . . . .	7		
B. Particulate Matter and Ash Contents of Petroleum Distillate Fuel . . . . .	8		
Particulate Matter Content Procedure . . . . .	8		
Ash Content Procedure . . . . .	9		

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glomerating into large particles that could clog filters; a **metal deactivator** to reduce the tendency of metals such as copper, brass, and zinc to accelerate sludge formation; a **corrosion inhibitor** to prevent internal tank corrosion; and a **biocide** to prevent microbiological growth in the fuel storage system. The inhibitor should preferably be added just prior to a fuel delivery to take advantage of the mixing that occurs as new fuel enters the tank. In large multitank fuel storage systems, in-house pumping systems can be used to mix the inhibitor with the fuel by valving (manually) to transfer fuel between tanks and introducing the required amount of inhibitor while the transfer is in progress.

1.04 At the initial fill and when fuel is added to a tank, the supplier should be cautioned not to introduce dirt into the storage system. If feasible, new fuel should be allowed to settle for at least 6 hours before it is used.

1.05 **DANGER: Keep open flames and sparks away from the fuel and the fuel tanks. Partially filled tanks and even empty tanks that have held fuel may contain explosive gases.**

## 2. APPARATUS

2.01 **List of Tools and Materials:** The following is a list of tools and materials needed.

TOOLS	DESCRIPTION
—	Dipstick, length as required
—	Sampling Kit, provided or suggested by testing laboratory
◆ —	Pump and Hose for Water Removal (may be included with sampling kit)
<b>MATERIALS</b>	
KS-21923, L1	Fuel Inhibitor or equivalent
KS-21527	Eyewash Solution or equivalent
—	Water Finder Paste◆

## 3. FUEL REQUIREMENTS

### A. Diesel and Gas Turbine Engines

3.01 ◆The preferred fuels for diesel engines are those conforming to the American Society for Testing and Materials (ASTM) Standard D975, Grades No. 1-D and No. 2-D as certified by the supplier. The preferred fuels for gas turbine engines are those conforming to the ASTM Standard D2880, Grade No. 1-GT. Grade No. 1-GT fuels will, in general, provide the best possible exhaust quality, the longest engine component life, and greater starting reliability.

**Note:** It is important for the reliability of the using system that the user specify that the cloud point of the fuel (temperature at which wax that could clog filters initially appears to be 6°C (11°F) below the lowest temperature to which the fuel will be subjected.

3.02 In emergencies, such as those due to disruption of normal supply sources, diesel fuel oil Grade No. 1-D or No. 2-D not known to be in compliance with the above specifications may have to be used in diesel engines. When normal sources of supply are reestablished, area supervision should be consulted with regard to replacement or treatment of any fuel found to be substandard.

3.03 Some gas turbine engines will operate satisfactorily on fuels conforming to ASTM D2880, Grade No. 2-GT. Before such fuels are used, however, it is recommended that the engine manufacturer be consulted.

3.04 **KS-20460 gas turbine-alternator sets are restricted to the use of fuels meeting the requirements of ASTM D2880, Grade No. 1-GT.** An additional requirement of a luminometer number greater than 40 must also be satisfied. Because of the present design of the combustion system of this set, engine life could be adversely affected by the use of fuels that do not meet these requirements.

3.05 Fuel inhibitor KS-21923, L1, should be used as covered in Part 7 for all fuels stored for use in diesel or gas turbine engine reserve power plants. This fuel inhibitor should not be used in gasoline.

## B. Gasoline Engines

**3.06** Only a limited number of stationary gasoline engines are still in use and the storage of fuel for these engines is therefore not covered by this section. The preferred fuel for gasoline engines is unleaded (white) gasoline. When engine starting becomes difficult because of carburetor gumming, a commercially available gum solvent should be used to clean out the carburetor.♦

## 4. RECOMMENDED FUEL STORAGE PRACTICES

### A. Maintenance Considerations

**4.01** ♦Fuel storage for reserve power plants requires more precautions than normal commercial fuel storage, primarily because of requirements for self-sufficiency in power generation during extended emergencies.

**4.02** Installed fuel storage capacity should generally be based on the anticipated essential system load, rather than on installed engine plant capacity and full load consumption rates. Fuel consumption rates at full and partial loads are available for all standard engine sets in the appropriate engine plant J-specification or section.

**4.03** Part 7 covers the use of fuel inhibitor to extend the useful life of stored fuels. The useful life of fuels tested and treated according to this section is expected to be at least 20 years.

**4.04** Steel fuel tanks should be kept as full of inhibited fuel as possible to prevent tank corrosion caused by condensed atmospheric moisture. Fiberglass tanks are not subject to corrosion, but the fuel stored in them should also be inhibited and maintained according to this section.

**4.05** It is obviously important to obtain a satisfactory grade of fuel that is free of contaminants and can be pumped and poured at the lowest temperatures to be encountered, both at the initial and at the subsequent fillings of the fuel storage system. At each fill, KS-21923, L1, inhibitor should be added to the tank at the rate of 1 gallon per 2000 gallons of uninhibited fuel being introduced. Following both initial and subsequent fillings, after the fuel has been allowed to settle in the tank for at least 6 hours, a sample should be obtained and analyzed. If the results of the analysis, interpreted according to the

guidelines of Table A, indicate that additional inhibitor is needed, the dosage called for in Table A should be added. Following this addition of inhibitor, but only after at least 48 hours have elapsed, the fuel should be resampled and the sample analyzed to assure that no further inhibitor addition is needed.

**4.06** Fuels in both storage and day tanks should be sampled and tested at least every 2 years.

### B. Laboratory Test Methods

**4.07** The recommended tests to be performed are listed below. The test methods are covered in detail in Part 9.

(a) Accelerated Aging [see Part 9(A)]

(b) Particulate Matter and Ash Contents [see Part 9(B)].

**4.08** A number of independent laboratories are capable of performing the tests called for in paragraph 4.07. The test methods of Part 9 should be provided to any laboratory being considered as a prospective supplier of such services.♦

## 5. WATER DETECTION AND REMOVAL

**5.01** ♦Free water (water that is not part of an emulsion with the fuel) will settle to the bottom of a fuel storage tank. Each storage tank should be checked for water accumulation at least once a year, and every tank must be checked prior to taking a fuel sample for testing. A certain amount of water may be expected to be found in any fuel storage tank as a result of the condensation of atmospheric moisture. The accumulation of water is an undesirable condition, and water should therefore be pumped out of tanks as often as practical. In any event, water must be pumped out before a fuel sample is taken for analysis, and it should be pumped out well before it reaches a depth of half the distance from the bottom of the tank to the level of the foot valve opening.

**5.02** *Warning: Do not drop the dipstick into the tank. Even though some tanks contain local reinforcement below a dipstick opening, the bottom of the tank could be damaged.* Water accumulation can be determined with a dipstick coated with water finder paste. At least the bottom 6 inches of the dipstick should be coated with water finder paste and the dipstick

should be inserted through the fill port or, if necessary, through some other access port until it touches the bottom of the tank. The instructions provided by the manufacturer of the water finder paste should be followed in applying the paste and in interpreting the color changes of the paste.

**5.03** Some fuel storage tanks have water drainoff piping built into them, and in some cases this pipe provides the only tank opening available for inserting the dipstick. In all cases, the water drainoff pipe must be removed and discarded before the amount of water in the bottom of the tank is measured using this access port.

**5.04** Water (with sludge and other material suspended in it) may be removed from the bottom of a tank using a pump and a flexible suction hose whose open end is either lowered to the tank bottom or maneuvered to the bottom secured to the end of the dipstick. The water and suspended material should be pumped into a clean container and saved for proper disposal. Pumping should continue until clear fuel oil appears. The volume of water removed and the date of removal should be recorded so as to call attention to unusually high rates of water accumulation.♦

## 6. FUEL SAMPLING

**6.01** ♦For best results, fuel samples should be taken from a point at or very near the bottom of the storage tank. A water drainoff pipe must not be used for sampling; but, if no other access port is available, the water drainoff pipe can be removed and discarded (as it must be before measuring water accumulation) and this port used for obtaining the fuel sample.

**6.02** The testing laboratory selected should be able to suggest a supplier of sampling kits and sample containers or will be able to provide them, together with information on the quantity of sample required for analysis. The laboratory may also provide assistance in sample taking, if this is desired; similar services are available from other companies. Because sampling kits are not standardized, the sampling procedures that follow are given in general rather than specific terms.

**6.03** *Sampling Procedure:* Use the following procedure to obtain fuel samples.

- (1) Check for water accumulation as in Part 5 and pump all water and material entrained in it from the bottom of the tank. Collect this in a clean container, record the volume of water removed and the date of the removal, and arrange for proper disposal. (It may be appropriate to incorporate the water removal procedure in the first stage of sample collection.)
- (2) Make sure that all the items needed for sampling are clean and on hand.
- (3) Lower the suction hose into the tank so that the open end of the hose reaches the tank bottom or is no more than 3 inches from it. This operation may be facilitated by taping the hose to the dipstick. (The end of the hose must be at the tank bottom if water is to be removed.)
- (4) Connect the suction pump, and, if water is to be removed, pump and collect the water as indicated in Step (1) until clear fuel appears. If it is not necessary to remove water from the tank, pump and collect fuel in a clean test container until the container is about half full to purge the collection system of any contamination. Then, with a swirling action, empty the test container into a waste fuel container for proper disposal. This fuel should not be returned to the tank.
- (5) Once clear fuel is being pumped after water is removed or the system has been purged, pump and collect somewhat more than the required amount of sample in a clean test container or the emptied container used for purging.
- (6) Transfer the fuel sample from the test container to the sample container furnished or specified by the testing laboratory, leaving at least 25 percent of the volume of the container empty to allow for vapor expansion. This will prevent bursting or leaking caused by internal pressure.
- (7) Place the insert and cap securely on the sample container.
- (8) Empty any remaining fuel from the test container into a waste fuel container for proper disposal, and dismantle and clean the sample kit.
- (9) Fill in the mailing label on the sample container, furnishing as much of the information shown on the typical label in Fig. 1 as possible. Be

sure to indicate on the label the tank and port from which the sample was taken.

**6.04 Alternate Sampling Procedure:** If (and only if) it is not possible to obtain a fuel sample by the method of paragraph 6.03, the following alternate procedure may be used.

- (1) Locate the first fuel filter in the supply line leading from the tank to be sampled (usually at the day tank or an engine).
- (2) If the filter element is the disposable type, obtain a new element.
- (3) Remove the filter canister and wipe it clean, replacing the filter element with a new one if it is the disposable type.
- (4) Use the pump that transfers the fuel to the filter to refill the canister and then drain it into a clean test container, repeating these operations until a sufficient amount of fuel is obtained for a sample.
- (5) Continue with Steps (6) through (9) of paragraph 6.03.
- (6) If the filter is located at an engine, test run the engine under load for at least 30 minutes to insure proper operation.
- (7) Contact the building supervisor and report the inaccessibility of an appropriate sampling port. Forward this information through proper channels so that the condition will be corrected.

**6.05 Sample Handling:** Fuel samples should be sent to the testing laboratory as soon as possible after the sample is taken, using a carrier who can assure rapid delivery. Fuel samples that are mishandled, particularly those that are subjected to elevated temperatures for extended periods of time, will not give meaningful test results.

**6.06 Test Results:** Table A provides guidelines on the actions to be taken based on the results of the laboratory tests.♦

## 7. FUEL INHIBITOR

**7.01 ♦DANGER:** *Although the fuel inhibitor vapors are not toxic, adequate ventila-*

*tion should be provided during its handling. Contact with the eyes, skin, and clothing should be avoided. In case of contact with the eyes, the eyes should be flushed with water or with KS-21527 eyewash solution for 15 minutes and then given medical attention. Although contact with the skin will generally not produce excessive irritation, such contact should be held to a minimum. In case of contact with the skin, the skin should be washed thoroughly with kerosene and then with soap and water. In case of contact with clothing, the clothing should be removed as soon as possible to minimize transfer to the skin. The clothing should be rinsed with kerosene (if this does not damage the material) and then washed with soap (detergent) and water.* The KS-21923, L1, inhibitor is a liquid solution of organic chemicals containing a stabilizer, a dispersant, a metal deactivator, a corrosion inhibitor, and a biocide. The inhibitor, when used in the recommended quantities, will not interfere with engine operation. Paragraph 4.05 deals with inhibitor additions at the time of fuel deliveries, and Table A provides guidelines on inhibitor additions based on the results of laboratory tests of fuel samples.♦

## 8. ON-SITE FUEL FILTRATION

**8.01 ♦** Fuel should be subjected to on-site filtration only when the concentration of solid inorganic material (sand, rust, etc.) in the fuel is such that engine operation would be placed in jeopardy. Elevated concentrations of organic particulate matter can generally be reduced to satisfactory levels by treatment with KS-21923, L1, fuel inhibitor, and elevated concentrations of inorganic particulate material (as measured by the ash content of the fuel) can usually be dealt with by simply pumping out the tank bottoms, where such particulates tend to collect.

**8.02** A number of companies offer on-site fuel filtration service using mobile pumping and filtration equipment. The fuel is pumped from the storage tank through filters and back into the storage tank. This recirculation is continued until the fuel is sufficiently clean. Pumping rates should be high enough to promote strong circulation in the tank to stir up material that has settled to the bottom and to displace loose material from the walls. The filters used should be capable of removing solid particles one micron or larger in diameter with an efficiency of at least 90 percent. It is desirable, if pos-

DATE OF SAMPLE \_\_\_\_\_ TYPE ENGINE: PISTON \_\_\_\_\_ TURBINE \_\_\_\_\_

SAMPLE PORT LOCATION \_\_\_\_\_

LOCATION CODE \_\_\_\_\_ TYPE FUEL: DIESEL \_\_\_\_\_ KEROSENE \_\_\_\_\_ GASOLINE \_\_\_\_\_

ADDRESS OF STORAGE TANK LOCATION \_\_\_\_\_

CITY \_\_\_\_\_ STATE \_\_\_\_\_ ZIP CODE \_\_\_\_\_

NO. GALLONS IN TANK WHEN SAMPLE TAKEN \_\_\_\_\_

PREVIOUS SAMPLE DATE \_\_\_\_\_ AREA \_\_\_\_\_ DISTRICT \_\_\_\_\_

DISTRICT OFFICE ADDRESS \_\_\_\_\_ ROOM NO. \_\_\_\_\_

CITY \_\_\_\_\_ STATE \_\_\_\_\_ ZIP CODE \_\_\_\_\_

Fig. 1 — Typical Sample Container Information Label

♦TABLE A♦		
GUIDELINES FOR INTERPRETING FUEL TEST RESULTS		
TEST	CONDITION	RECOMMENDED ACTION
Accelerated Aging [see Part 9(A)]	<b>Stability No.</b> 7.0 or less 8.0 to 10.0 11.0 to 15.0 over 15.0	None Add 1 gal KS-21923, L1, per 2000 gal fuel Add 1 gal KS-21923, L1, per 1000 gal fuel Add 1 gal KS-21923, L1, per 500 gal fuel*
Particulate Matter [see Part 9(B)]	>2.0 mg/100 cm <sup>3</sup> of sample	Determine Ash Content
Ash Content [see Part 9(B)]	>1.0 mg/100 cm <sup>3</sup> of sample	Resample and confirm before filtering. If filtering is required, resample and confirm reduction in ash content after filtering.

\*After 48 hours, resample and determine stability rating. If the stability number is greater than 7.0, reinhibit according to the above schedule.

sible, to have laboratory studies carried out to demonstrate that on-site filtration will reduce the particulate concentration to a satisfactory level before it is undertaken.

**Note:** It should rarely be necessary to filter fuel, and it should never be necessary to discard it.▲

## 9. DESCRIPTION OF FUEL TEST PROCEDURES AND LABORATORY METHODS

**9.01** This part gives recommended test methods and procedures to be used by the testing laboratory.

### A. ▲ Accelerated Aging of Petroleum Distillate Fuel

**9.02** *Outline of Method: DANGER: Test specimens and solvent constitute hazardous substances with regard to combustion, health, and disposal. The test procedure should be conducted with appropriate precautionary measures. At each location, existing in-house safety and disposal regulations should be followed in every detail.* A 50 cubic centimeter sample of the fuel is filtered, heated, cooled, and filtered again. The final filter is compared to a set of standards or measured with a light transmission instrument to obtain a numerical rating of stability.

**9.03** **Applicability:** This method is applicable to all petroleum distillate fuels.

**9.04** **Apparatus and Materials:** The following apparatus and materials are required to perform this procedure.

(a) **Vacuum Filtration Apparatus:** Use 42.5 millimeter diameter Whatman No. 1 filter paper discs or equivalent in either a Buchner funnel or a Millipore filter holder. The Millipore filter holder is available under the name Pyrex\* Filter Holder, catalog number XX1C 047 00, from Millipore Corporation, Bedford, Massachusetts 01730, 617-725-9200.

(b) **Oil Bath:** The oil bath shall be capable of operating at  $149 \pm 2.8^{\circ}\text{C}$  ( $300 \pm 5^{\circ}\text{F}$ ) and

\* Trademark of Corning Glass Works, Houghton Park, Corning, N.J.

shall be filled with silicone oil. An appropriate oil is DC550, which is available from Dow-Corning Corporation, Midland, Michigan 48640, 800-248-2345.

(c) **Standard Reference Chart:** This chart is a part of duPont Method No. F21-61 and is available from Petroleum Chemicals Division, E. I. duPont de Nemours and Company, Wilmington, Delaware 19898.

(d) **Fuel Photometer:** This instrument uses a light transmission technique to determine the stability rating of a fuel and is available from Jantzen Products, Inc., 190 Parish Drive, Wayne, New Jersey 07470, 201-696-1003.

(e) **Petroleum Ether:** This solvent shall have a boiling range of 30 to 60°C.

**9.05** **Procedure:** The entire procedure must be carried out in a fume hood. To determine the stability rating of a fuel by accelerated aging, perform the following steps:

(1) Set up for filtration in the fume hood, and filter 50 cubic centimeters of the well-shaken fuel sample using a vacuum of approximately 16 inches of mercury.

(2) Pour the filtrate into a 2.5 by 20 centimeter test tube and place the tube in the oil bath for  $90 \pm 2$  minutes. The oil bath must be maintained at  $149 \pm 2.8^{\circ}\text{C}$  ( $300 \pm 5^{\circ}\text{F}$ ).

(3) Remove the test tube from the oil bath and allow it to cool to room temperature.

(4) Filter the specimen as in Step (1) using a new filter paper disc. Wash the residue remaining on the filter with petroleum ether and allow it to dry.

(5) Compare the dry filter with the standard reference chart or insert it into the fuel photometer. Read the stability number.

(6) The stability rating ranges from No. 1 to 20. Samples with stability ratings of No. 7 or lower are generally considered as passing the test.

**Note:** Do not flush the sample fuel, filtrate, or washings into the sewer system. Retain them in a safety can for noncontaminating disposal.

## B. Particulate Matter and Ash Contents of Petroleum Distillate Fuel

**9.06 Outline of Method:** **DANGER:** The test specimens and solvent constitute hazardous substances with regard to combustion, health, and disposal. The test procedure should be conducted with appropriate precautionary measures. At each location, existing in-house safety and disposal regulations should be followed in every detail. A 100 cubic centimeter sample of the fuel is filtered, and the residue is dried and weighed. The total particulate matter content is calculated, and, as necessary, the ash content is determined from the weight of the residue after ignition.

**9.07 Applicability:** This method is applicable to the determination of the particulate matter content and the ash content of all petroleum distillate fuels. It utilizes a gravimetric procedure considered to be convenient in the evaluation of fuels stored for use in auxiliary power generating equipment. Where a more precise method is desired, refer to ASTM D2276 Methods A and B.

**9.08 Apparatus and Materials:** The following apparatus and materials are required to perform this procedure.

- (a) **Vacuum Filtration Apparatus:** Use Millipore MF Type AA 0.8 micron nominal pore size cellulose filter discs in a Millipore Pyrex Filter Holder (catalog number XX1C 047 00). The filter discs and holder are available from Millipore Corporation, Bedford, Massachusetts 01730, 617-275-9200.
- (b) **Noncirculating Oven:** The oven shall be capable of operating at  $90 \pm 5^\circ\text{C}$ .
- (c) **Muffle Furnace:** The muffle furnace shall be capable of operating at  $750 \pm 30^\circ\text{C}$ .
- (d) **Petroleum Ether:** This solvent shall have a boiling range of 30 to  $60^\circ\text{C}$ .

## Particulate Matter Content Procedure

**9.09** To determine the particulate matter content, perform the following steps:

- (1) Pour the fuel to be tested into a beaker, taking care to include any residue that may lie at the bottom of the sample container by rinsing the container with some of the sample. Divide the fuel to be tested (300 to 400 cubic centimeters) into portions of 100 cubic centimeters.
- (2) Carry out the following steps for at least three of the 100 cubic centimeter specimens. With a noncontaminating marker, mark the rim of a filter disc for each of the specimens, and mark at least one additional filter to be used as a control. The control filter is to be carried through every stage of the procedure except for the introduction of a test specimen.
- (3) Handle filters only with tweezers. Place the filters in a covered glass petri dish and heat in the oven at  $90 \pm 5^\circ\text{C}$  for 30 minutes with the cover slightly ajar. Remove and cool in a desiccator to room temperature.
- (4) Weigh the dried filters individually to an accuracy of 0.1 milligram on an analytical balance and record the weight ( $W_0$ ) of each.
- (5) Place a dried and weighed filter on the filter holder and clamp the funnel to the filter holder. Pour the entire test specimen associated with the filter into the filter funnel, and apply a vacuum to the filter flask to draw the specimen through the filter. Maintain the vacuum at 13 to 15 centimeters (5 to 6 inches) of mercury.
- (6) During filtration, rinse the specimen container and the funnel with at least 100 cubic centimeters of petroleum ether. When the specimen and the petroleum ether have passed through the filter, remove the funnel and, with the vacuum still applied, carefully rinse the surface of the filter with the solvent. Rinse the accumulation of sediment toward the center of the filter. Continue the vacuum for 5 minutes after all of liquid has passed through the filter.
- (7) Release the vacuum, and, using tweezers, carefully remove the filter and place it in a covered petri dish. Leaving the cover slightly ajar, place

the petri dish in the oven at  $90 \pm 5^\circ\text{C}$  for 30 minutes. Remove and cool in a desiccator to room temperature.

(8) First weigh the control filter and then the test filters containing the total particulate contaminant, recording the weight ( $W_1$ ) of each. Reweigh the control filter to check that all weighings were conducted under comparable conditions. If the two weighings of the control filter do not agree, repeat the series of weighings until they do.

(9) Subtract the initial weights ( $W_0$ ) of the filters from their final weights ( $W_1$ ). If the gain in weight for the control filter is greater than 0.5 milligram, this indicates that residual petroleum ether remains on the filters, and they should be reheated as in Step (7) and reweighed as in Step (8).

(10) The particulate matter content ( $W_2$ ) is determined as the gain in weight of the test filter discs ( $W_1 - W_0$ ). See Table A for guidelines for interpreting the results.

**Note:** Do not flush the sample fuel, filtrate, or washings into the sewer system. Retain them in a safety can for noncontaminating disposal.

#### Ash Content Procedure

**9.10** If the particulate matter content is greater than 2.0 milligrams per 100 cubic centimeters of sample, it is desirable to determine the ash content. To determine the ash content, perform the following steps:

(1) Clean a crucible and fire it in the muffle furnace at  $750 \pm 30^\circ\text{C}$  for 10 minutes. Cool the crucible to room temperature in a desiccator and weigh it to an accuracy of 0.1 milligram on an analytical balance, recording the weight ( $W_3$ ).

(2) Place a filter disc with its particulate matter residue in the weighed crucible, and soak it with 1 to 2 cubic centimeters of mineral oil. Burn the filter disc and its residue in the crucible for 3 to 5 minutes, and then ignite the crucible and residue in the muffle furnace at  $750 \pm 30^\circ\text{C}$  for 20 minutes. Cool the crucible to room temperature in a desiccator and weigh it, again recording the weight ( $W_4$ ).

(3) The ash content ( $W_5$ ) is determined as the gain in weight of the crucible ( $W_4 - W_3$ ). See Table A for guidelines for interpreting the results.♦