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A FIELD MONITOR FOR THE STABILITY AND CLEANLINESS OF DISTILLATE FUEL

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By

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This report details the development of a portable device to measure the cleanliness and stability of diesel fuels in both bulk and vehicle fuel cell storage. Both an initial laboratory prototype and a portable field unit are discussed. The monitor evaluates the cleanliness of the fuel by first filtering the fuel and then determining the amount of particulates on the filters using a contaminated filter measuring device. The filters are rated by measuring the amount of light which passes through the filters

20. ABSTRACT (cont'd)

to estimate the amount of particulates based on a calibration chart. A measure of the stability of the fuel is obtained by aging a sample of the fuel at 150°C for 1.5 hours and determining the amount of sediment formed. Based on preliminary correlations of 150°C test data with ASTM D 2274 accelerated stability data, "go/no-go" values of <4,4-6, and >6 mg/100 ml have been suggested for rating a fuel's stability as good, marginal, or bad, respectively. Improvements to the diesel fuel quality monitor have been identified and are being investigated. A field test program to demonstrate monitor utility/effectiveness is planned for initiation in late 1981. In addition, a commercially available portable unit to measure particulate contamination is described and compared to the Army field test unit.

FOREWORD

The work presented herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (USAFLRL), Southwest Research Institute, San Antonio, Texas, under Contracts DAAK70-78-C-0001, DAAK70-80-C-0001, and DAAK70-82-C-0001. The work was funded by the U.S. Army Mobility Equipment Research and Development Command (USAMERADCOM), Ft. Belvoir, VA. Contracting Officer's representative was Mr. F.W. Schaekel, and the technical monitor was Mr. M.E. LePera, Fuels and Lubricants Division, Energy and Water Resources Laboratory (DRDME-GL).

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I. INTRODUCTION

Long-term storage stability and cleanliness have been requirements imposed on military fuels designed for tactical/combat environments. These requirements have been necessary because of a need to ensure that fuels delivered or deployed in strategic locations will be satisfactory for use during combat operations regardless of the time interval between refinery production and ultimate use. This becomes very critical in relation to prepositioning of fuel stocks in which good fuel quality must be maintained. Without guaranteed maintenance of fuel cleanliness and stability, combat fuels could prove to be the weak link during combat operations, leading to engine malfunctions, increased maintenance problems, equipment vulnerability, decreased mobility, and numerous other problems.

Table 1, listing major thrust areas which have been identified in the Army's distillate fuel stability program, has provided the basis for a discussion of Army needs for diesel fuel stability and cleanliness in a report distributed in 1980.(1)*

TABLE 1. MAJOR THRUST AREAS IN ARMY DISTILLATE FUEL STABILITY PROGRAM

Thrust Areas	Purpose
•Field Test Technique Development	Test quality and predict future quality
•Survey Quality of Petroleum Fuels	Correlate laboratory methods, specifications, field manuals
•Conduct Field Problem Surveillance	Identify fuel-related problems and adequacy of fuel procurement and handling practices
•Evaluate Additives for Depot Use and Prepositioning	Provide fuel additive package for dormant storage of fully fueled vehicles
•Conduct Basic Research	Define factors leading to deleterious products in both short- and long-term storage

The current report details the development of a field fuel quality monitor for the stability and cleanliness of distillate fuel to address the first thrust area, "Field Test Technique Development," given in Table 1.

II. BACKGROUND

Quick, simple, and most importantly, reliable methods of detecting excess fuel contamination while in the field have long been sought. Field laboratories offer a solution to this problem, but are usually large (requiring a vehicle to transport them) and expensive to operate and maintain. A more practical solution is a small, self-contained portable kit, which would be used to furnish a go/no-go type answer with regards to any given supply of fuel. Kits of this type are available commercially.

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*Superscript numbers in parentheses refer to references listed at the end of this report.

One such device is the Fuel Contamination Detector (NSN: 2H6630-706-2302).(2) This kit has a self-contained vacuum pump to filter the fuel, and uses a photovoltaic cell to determine the amount of light that passes through the filter as a measure of the amount of contamination. Although the unit was developed for turbine fuel and has not been fully evaluated for diesel fuel, the Navy has evaluated it for use with marine diesel fuel, concluding that the device could be used to give a go/no-go answer.(3) A major drawback of the kit is that it requires an a/c power source.

The Army has developed a Fuel Contamination Test Kit.(4) The kit, designed primarily for field use, is used to determine both the free (undissolved) water content and particulate content of liquid aviation and automotive fuels. It is also used to identify liquid fuel types based on gravity. In order to determine particulates, a known volume of fuel is filtered through two matched-weight membrane filters (test and control) contained in a plastic holder. The filters are then removed, dried, and weighed on a torsion balance. Particulate content is determined by comparing the increase in weight of the test membrane over the control membrane. This kit has not yet been fielded for use by the Army. One drawback is that care must be taken to level and calibrate the kit's balance; and after leveling and calibration, it should be moved as little as possible. Also, the kit requires the use of matched weight filters, which are costly relative to unmatched filters.

Another test used in the past for determining fuel cleanliness is the Filterability Ratio Test (see Appendix A). In this test, the time taken to filter a given amount of contaminated fuel is compared to the filtering time for an equal portion of the same fuel that has been prefiltered. A value of 1.0 for the test indicates a perfectly clean fuel. However, this test requires that the vacuum source be constant, which is sometimes difficult in a field situation. This procedure has been used with considerable success in the laboratory in predicting potential fuel deterioration/cleanliness problems.

In addition to testing for cleanliness, it is desirable to rate a fuel's approximate storage stability. The U.S. Army Fuels and Lubricants Research Laboratory (AFLRL) has an ongoing program devoted to the development and evaluation of an in-the-field fuel cleanliness/stability testing device.

A laboratory study was initiated in late 1976 to define the relative stability characteristics of a base set of fourteen test fuels. This study was used as input to the definition of a field test capability for measuring quality and predicting stability. Table 2 summarizes particulates data for seven test fuels

TABLE 2. DISTILLATE FUEL STABILITY DATA BASE—
PARTICULATES BY VARIOUS ACCELERATED STORAGE TESTS

Stability Test	Particulates, mg/100 ml, D 2276 (mod., 1.2μm) Fuel Sample Number						
	1	2	3	4	5	6	7
•D 2274	0.2	0.3	0.3	0.7	1.1	1.8	6.3
•D 873	0.4	1.6	1.2	0.9	2.8	5.1	19.9
•150°C, 90 min	1.0	3.4	1.0	1.6	2.0	2.6	4.3
•80°C, 72 hr	0.8	1.2	0.3	0.4	0.4	1.7	1.4
168 hr	0.6	1.7	0.4	1.0	1.7	3.5	4.1
•43°C, 16 weeks	0.8	0.6	1.1	0.2	0.2	3.6	0.4
32 weeks	0.8	0.5	1.1	0.5	0.2	5.5	1.4

using five accelerated test procedures described in Appendix A. Note that the particulates in the 150°C, 80°C, and 43°C methods were determined using ASTM Method D 2276, modified to incorporate a 1.2-micrometer pore size membrane filter.

Based on data correlations of the fuels ranked according to particulates and sediment at 43°C (110°F) storage for 32 weeks, the best correlations were with the 80°C (175°F) test data. The D 2274 test data correlation was particularly poor. The test with the greatest potential for field use, based on its simplicity and short test time, was the 150°C (300°F) test tube method requiring 90 minutes of reaction time. Unfortunately, the 150°C test data correlated poorly for the ranked fuels. However, this method may offer some potential if a go/no-go value of 4.0 mg/100 ml is used. The data given in Table 3 for a number of field samples, some of which are from vehicle fuel cells, reinforce the suitability of the 4.0 mg/100 ml value for go/no-go determination of fuel quality.

TABLE 3. COMPARISON OF ASTM METHOD D 2274
AND 150°C TEST DATA FOR FIVE FUELS

	Particulates, mg/100 ml				
Method	Fuel 1*	Fuel 2*	Fuel 3*	Fuel 4**	Fuel 5**
D 2274	4.9	3.2	3.2	5.7	0.9
150°C, 90 min	11.3	8.6	6.0	10.2	2.6

* Vehicle fuel cell samples
** Bulk underground storage samples

Federal specification VV-F-800C for diesel fuel limits the D 2274 value to 1.5 mg/100 ml total insolubles; hence, the values for fuels 1 through 4 in Table 3 are seen to be high both with respect to D 2274 and the test at 150°C for 90 minutes. The values in Table 4 were recently presented (5) as

TABLE 4. FUEL STABILITY RATINGS OF GOOD, MARGINAL, AND BAD
BASED ON LIMITING INSOLUBLES VALUES (3)

	Limiting Values for Rating Fuels (mg/100 ml, glass fiber filters)		
Test Method	Good	Marginal	Bad
D 2274	<1.5	1.5-2.2	>2.2
43.3°C Vented			
4 wk	<2	2-3	>3
8 wk	<2	2-3	>3
16 wk	<2	2-3	>3
24 wk	<2	2-3	>3
150°C			
1.5 hr	<4	4-6	>6
3.0 hr	<4	4-6	>6
4.5 hr	<4	4-6	>6
80°C			
3 days	<2	2-3	>3
7 days	<2	2-3	>3
14 days	<4	4-6	>6
D 873	<4	4-6	>6
93.3°C	<1.5	1.5-2.2	>2.2

go/no-go values based on test results for six fuels in a program to develop accelerated stability test techniques for middle distillate fuels. For purposes of judging inherent fuel stability, total insolubles limiting values were selected (Table 4) for each of the six test methods. Fuels then can be rated as good, marginal, or bad if their insoluble values are less than the selected limiting value, less than 150 percent of the limiting value, or greater than 150 percent of the limiting value, respectively. Note that the limiting values for good, marginal, and bad for the 150°C test are <4, 4-6, and >6 mg/100 ml total insolubles, respectively.

This report details the development of an initial laboratory prototype and a field prototype fuel quality monitor, and discusses a commercially available unit for measuring fuel quality in the field.

III. LABORATORY PROTOTYPE FUEL QUALITY MONITOR

There are basically four functions which were envisioned that an in-the-field fuel cleanliness/stability testing device (fuel quality tester) should perform:

(1) sample the fuel from its storage container,
(2) rate the fuel for cleanliness,
(3) age the fuel in some manner, and
(4) rate the aged fuel for stability.

Methods of performing these functions were first evaluated in laboratory approaches prior to fabricating a field test unit.

A. Experimental

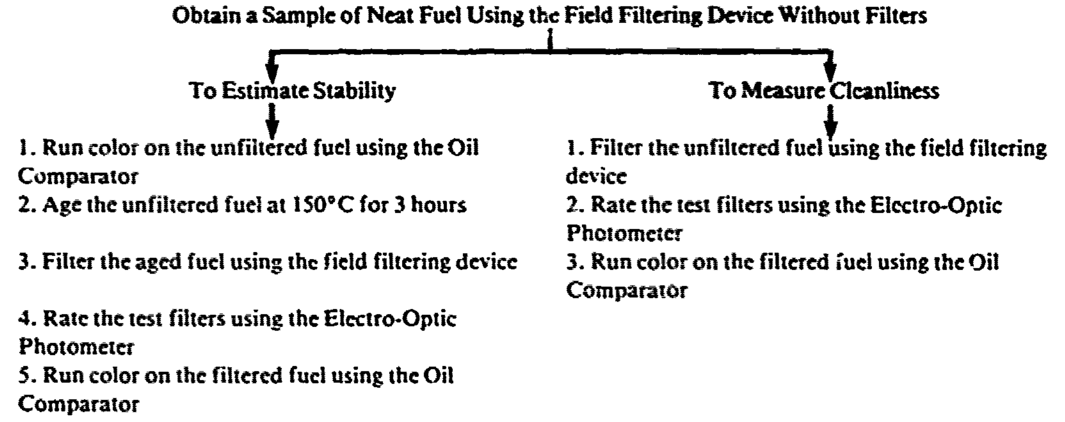
The laboratory prototype fuel quality tester was evaluated using nine fuels which had been in ambient, vented drum storage for 3 years. The 55-gallon drums containing the test fuels were each moved to a shaded area and stirred for 15 minutes with an air-driven stirrer. The drums were allowed to stand for 2 days prior to sampling to allow the water to settle out. Approximately 4 gallons of each fuel were sampled into a 5-gallon can. Each 5-gallon can was then used to simulate in-the-field fuel storage containers. Some of the unfiltered fuel was sent to the laboratory for standard tests, which included color, steam jet gum, water and sediment, total acid number, light absorbance, accelerated stability by ASTM Method D 2274, and particulates by ASTM Method D 2276 (modified). In addition, the filters from the D 2276 test were also rated using the prototype filter testing device. Table 5 is a compilation of this laboratory data. Examination of this data shows that the fuels used covered a wide range of stability, as measured by ASTM Method D 2274, but a more narrow range of cleanliness.

Figure 1 is a flow chart for the evaluation procedure. Note that there are two categories under which the evaluation procedures fall: "To Estimate Stability" and "To Measure Cleanliness". The device pictured in Figure 2 was used to obtain a sample of the neat fuel. This device consisted of a length of plastic tubing long enough to sample at the desired depth in the fuel container, a filter holder, a receiving flask, and a small hand pump used as a vacuum source. A hand-operated vacuum pump performed very well as the vacuum source and was able to create enough suction to sample the fuels from their storage containers. Note that no filters were placed in the filter holder while obtaining a sample of the neat fuel. To measure the cleanliness of the fuel, filters were installed in the field filtering device. The filters used during evaluation of the initial laboratory prototype device were 1.2-

TABLE 5. AMBIENT STORAGE OF DISTILLATE FUELS DATA AFTER 36 MONTHS STORAGE

Sample Code No.	6574	6624	6625	6626	6630	6638	6678	6716	6746
Particulates, D 2276, 1.2 μm, mg/500 ml	4.78	13.10	1.00	1.36	15.58	0.04	3.68	5.22	1.10
Accelerated Stability, D 2274, Total Insolubles, mg/100 ml	7.4	0.6	2.0	0.2	2.1	0.1	1.0	1.5	0.3
Steam Jet Gum, D 381, mg/100 ml	11.4	32.2	4.0	4.2	40.3	0.2	7.9	13.2	2.2
Color (D 1500)	1.0	3.0	1.0	2.0	6.0	0.5	4.0	3.0	0.5
Total Acid No., D 664, mg KOH/gm sample	0.026	0.140	0.085	0.009	0.040	0.016	0.085	0.039	0.063
Light Absorbance at 650 nm	0.021	0.159	0.001	0.015	0.292	0.001	0.075	0.041	0.001
575 nm	0.048	0.259	0.010	0.060	0.645	0.001	0.185	0.116	0.001
540 nm	0.073	0.346	0.026	0.107	1.132	0.001	0.372	0.193	0.001
500 nm	0.128	0.520	0.059	0.191	2.070	0.001	0.831	0.327	0.007
Water & Sediment (D 1796)	A	B	None	None	C	None	None	A	None

A = trace sediment; no water.
B = trace sediment; 0.30 vol% water.
C = 0.1 vol% sediment; no water.



Obtain a Sample of Neat Fuel Using the Field Filtering Device Without Filters

To Estimate Stability

1. Run color on the unfiltered fuel using the Oil Comparator
2. Age the unfiltered fuel at 150°C for 3 hours
3. Filter the aged fuel using the field filtering device
4. Rate the test filters using the Electro-Optic Photometer
5. Run color on the filtered fuel using the Oil Comparator

To Measure Cleanliness

1. Filter the unfiltered fuel using the field filtering device
2. Rate the test filters using the Electro-Optic Photometer
3. Run color on the filtered fuel using the Oil Comparator

FIGURE 1. FLOW CHART FOR EVALUATION OF FIELD FUEL QUALITY MONITOR

micrometer pore size cellulose acetate-cellulose nitrate membrane filters. The filter holder was manufactured by Schleicher and Schuell, Inc., model no. FP050/9.

Five hundred milliliters of the unfiltered fuel were filtered through two sandwiched 1.2-micrometer millipore filters (a sample filter and a control filter) using the prototype field filtering device (Figure 2). These filters were preweighed so they could be reweighed at a later time for gravimetric quantitation of particulates.

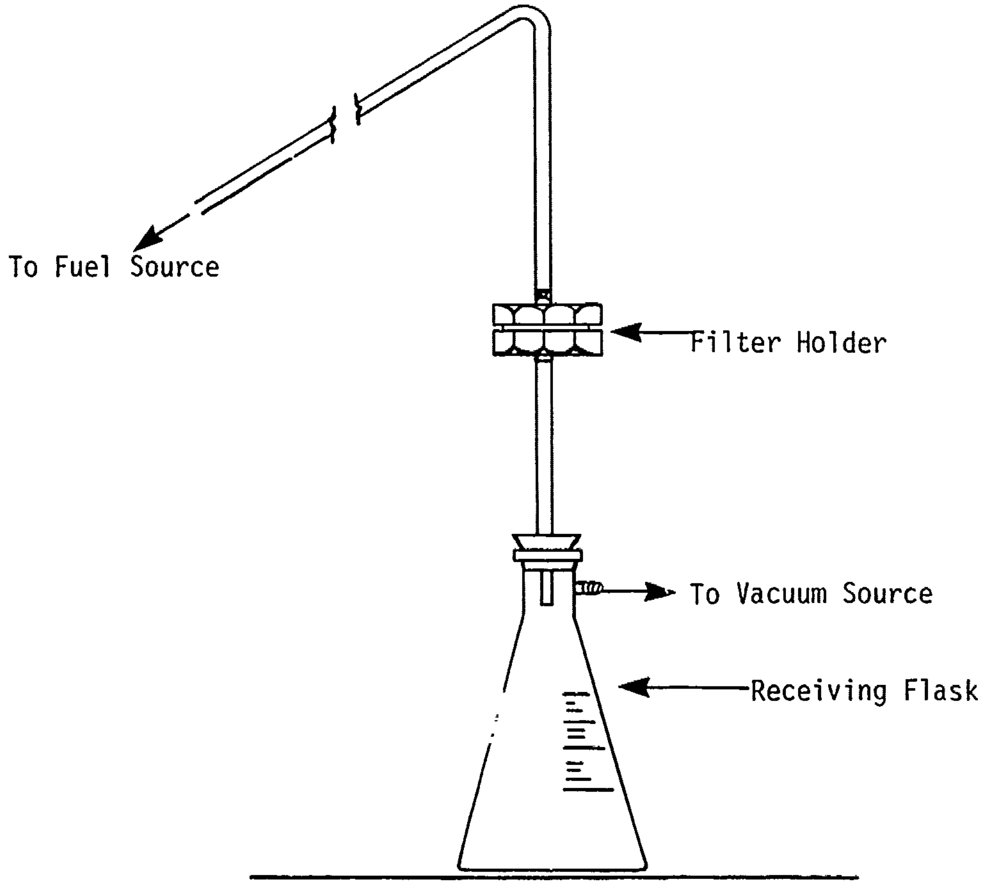


FIGURE 2. PROTOTYPE FIELD FILTERING DEVICE

To rate the filters for cleanliness, an electro-optic photometer (EOP) was breadboarded, feasibility having been qualitatively demonstrated previously. This instrument (Figure 3) shines a beam of light through the test filter and measures the amount of light that passes through the filter by means of a photosensitive cell. The lamp and detector of the EOP are shielded from external light during measurements.

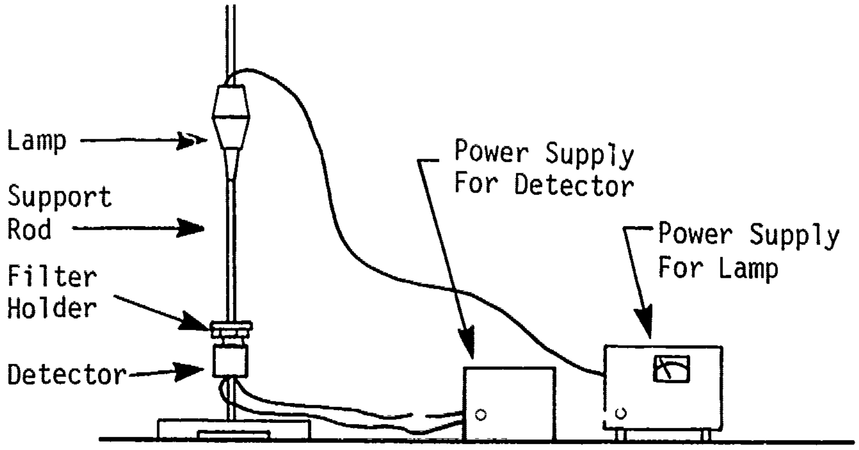


FIGURE 3. DIAGRAM OF LABORATORY PROTOTYPE ELECTRO-OPTIC PHOTOMETER APPARATUS

The amount of light that is blocked from passing through the filter is a function of the amount of particulate contamination present on the filter. To measure the color of the fuel, a device manufactured by Hellige Incorporated called a Hellige Oil Comparator Outfit No. 607-OC was used. This device allows for the comparison of the color of the fuel to a set of standard colored discs. Detailed operating instructions are found in Appendix B. To estimate the stability of the fuels, 55 milliliters of each fuel were prefiltered and placed into each of four test tubes to be heated at 150°C for 3 hours. After aging, the fuel in two of the tubes was filtered using the field filtering device (see Figure 2) with two preweighed 1.2-micrometer membrane filters, and the fuel in the other two tubes was filtered by modified ASTM Method D 2276, also using 1.2-micrometer membrane filters. The field filtering device test filters and the D 2276 filters were then rated using the EOP. The filtered fuel from the field filtering device was tested for color. The 1.2-micrometer filters from the D 2276 determination (both the 3-year storage samples and the 150°C aged fuel) were rated by the EOP in a dry state. The field filtering device filters were rated while still wet with fuel.

B. Discussion

The hand pump used with the field filtering device was able to create enough suction to filter most of the fuels. However, if water was present in the fuel, filter plugging often occurred. In some cases, when using the field filtering device, the particulates tended to localize on a given area of the filter. There was also a tendency of the fuel to flow either around the filters or between them because the filter holder did not hold the filters securely in place and the vacuum was not constant. Each of these occurrences can lead to erroneous readings when the filters are rated using the EOP. Table 6 lists data for the filtration of fuel through the field filtering device from both the filtration of the neat fuel and the filtration of the aged samples from the 150°C test. The table gives the weight of particulates left on each filter following filtration.

TABLE 6. DATA FOR FILTRATION OF FUEL THROUGH THE FIELD FILTERING DEVICE
(1.2-micrometer pore size membrane filter)

Sample Code No.	6574	6624	6625	6626	6630	6638	6678	6716	6746
Neat Fuel									
Weight of Particulates, mg/500 ml									
Sample filter	4.74	*	1.04	0.46	19.32	10.04	12.66	15.64	11.38
Control filter	1.46	*	1.00	0.08	12.66	10.02	12.00	8.14	10.08
Difference	3.28	*	0.04	0.38	6.66	0.02	0.66	7.50	1.30
150°C (3 hr Aging of Fuel)									
Weight of Particulates, mg/100 ml									
Sample filter	13.96	6.00	3.08	1.28	9.20	0.12	4.12	25.20	1.44
Control filter	0.12	0.24	0.08	0.28	0.60	0.12	0.00	0.28	-0.06
Difference	13.84	5.76	3.00	1.00	8.60	0.00	4.12	24.92	1.50

* = Sample would not filter due to high percentage of water present in fuel.

The EOP uses attenuation of a light beam by the test filter to estimate the amount of particulate contamination present on the filter. The amount of light that passes through the test filter is measured by a photosensitive cell. The percent light transmittance (%T) through each test filter was calculated using the formulas given in Table 7. The absorbance of each filter (sample and control) was calculated using the calculated transmittance values.

Particulate absorbance, the absorbance due to the particulate on the sample filter, is determined by subtracting the absorbance of the control filter from the absorbance of the sample filter.

TABLE 7. TRANSMITTANCE/ABSORBANCE CALCULATIONS FOR TEST FILTERS

Transmittance, T, is defined as

$$T = I_T/I_o$$

where

I_o = Initial Intensity

I_T = Transmitted Intensity

%T = T(100)

Absorbance, A, is defined as

$$A = \log 1/T = \log I_o/I_T$$

Particulate Absorbance is equal to A of sample filter minus A of control filter

The presence of particulates on the control filter (second or backup filter in each set) indicates that fuel was allowed to flow between the two filters. When this control filter weight is subtracted from the sample filter weight, the result is an erroneous weight for the total amount of particulates. Since the EOP reads the amount of particulates on the filter, the EOP readings would also be misleading if fuel were allowed to flow between the filters. One possible way to alleviate this problem is to use a filter holder much like the type used in the ASTM Method D 2276 in which the two parts of the holder are clamped together, rather than the type which screws together. Another solution might be to use a standard vacuum pump that has been converted to run by one or two 12-volt batteries as opposed to using a hand pump. This vacuum pump would conceivably deliver a greater and more constant vacuum and thus reduce the chances of the two filters separating and allowing fuel to flow between them.

For evaluation and calibration purposes it was necessary to determine the relation of the EOP results to the actual weight of the particulates on the filter. EOP readings were recorded for each filter, and absorbance values were calculated. These absorbances were then converted to particulate absorbance values (Table 7) and compared to the weight of particulates as determined gravimetrically. Table 8 is a listing of the various particulate absorbance values compared to the gravimetrically determined particulate weights.

Examination of the data in Table 8 shows that overall the particulate absorbance values for the samples filtered by D 2276 are higher for corresponding samples than those for the samples filtered using the field filtering device. This could be that the D 2276 filters were rated by the EOP while dry whereas the field filtering device filters were rated while still wet with fuel. Also, should the field filtering device allow fuel to flow around and between the filters, the results would be lower in value than they should be. Most likely the explanation is a combination of the above two reasons. Note also that the particulate weights for corresponding samples tend to be higher for the D 2276 filters. This is probably also a result of the fuel flowing around and between the filters. It was also noted that color bodies present in the fuel tended to adsorb onto the cellulose acetate-cellulose nitrate filter medium. These color bodies could absorb light but would not register as increased weight, thus leading to erroneous results.

Since the accuracy of the absorbance data obtained from the field filtering device filters was considered somewhat questionable, it was decided to use only the D 2276 filters for calibration of the EOP. Figure 4 is a plot of the particulate absorbance versus the particulate weight for the D 2276 filter data from Table 8. Examination of the data shows, with few exceptions, an increase in particulate absorbance as particulate weight increases. For this reason, it was felt that the EOP was a viable approach to estimating the amount of particulates on a filter.

TABLE 8. COMPARISON PARTICULATE ABSORBANCE VALUES TO ACTUAL PARTICULATE WEIGHTS

Results for Samples Filtered Using the Field Filtering Device

Neat Fuel, 1.2 μm			150°C (3 hr aging), 1.2 μm		
Sample No.	Particulate Absorbance	Particulate Weight, mg	Sample No.	Particulate Absorbance	Particulate Weight, mg
6574	0.1	3.28	6574	1.0	13.84
6624	*	*	6624	0.4	5.76
6625	0.1	0.04	6625	0.1	3.00
6626	0.1	0.38	6626	0.1	1.00
6630	0.7	6.66	6630	1.0	8.60
6638	0.1	0.02	6638	0.1	0.01
6678	0.6	7.50	6678	0.1	4.12
6716	0.1	1.30	6716	1.3	24.92
6746	0.2	0.66	6746	0.1	1.50

Results for Samples Filtered Using ASTM Method D 2276 (Modified)

Neat Fuel, 1.2 μm			150 C (3 hr aging), 1.2 μm		
Sample No.	Particulate Absorbance	Particulate Weight, mg	Sample No.	Particulate Absorbance	Particulate Weight, mg
6574	0.7	4.8	6574	0.5	7.4
6624	0.8	13.1	6624	1.1	5.8
6625	0.01	1.0	6625	0.2	3.4
6626	0.1	1.4	6626	0.1	1.2
6630	1.9	15.6	6630	1.6	11.1
6638	0.1	0.1	6638	0.1	0.1
6678	0.6	3.7	6678	0.5	5.0
6716	0.7	5.2	6716	1.7	25.8
6746	0.1	1.1	6746	0.1	1.2

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*Filter plugged by water in fuel

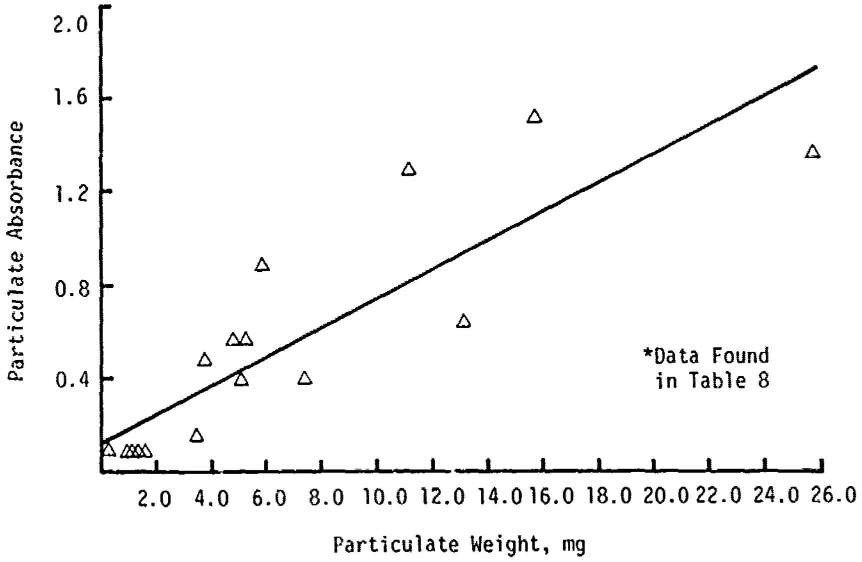

FIGURE 4. PLOT OF PARTICULATE ABSORBANCE VERSUS PARTICULATE WEIGHT
(Samples filtered using ASTM Method D 2276, mod., 1.2 μm)

To measure color, the Hellige oil comparator device was chosen because it is small, portable, easy to use, and requires no light source other than the sun. During the evaluation, color was run both by ASTM Method D 1500 and the oil comparator. The results of these color evaluations are found in Table 9. The oil comparator was found to give results equivalent to D 1500. As can be seen by the data, the values for D 1500 sometimes varied from the oil comparator values by 0.5 units; however, this slight deviation is within the precision of the D 1500 method. The operating instructions for the oil comparator are given in Appendix A.

TABLE 9. DETERMINATION OF COLOR BY ASTM METHOD D 1500 VERSUS THE HELLIGE OIL COMPARATOR

Sample Code No.	6574	6624	6625	6626	6630	6638	6778	6716	6746
Neat Fuel									
D 1500	1.0	3.0	1.0	2.0	6.0	0.5	4.0	3.0	0.5
Oil Comparator	1.5	3.5	1.0	2.5	6.5	0.5	4.0	3.0	0.5
D 2274 Filtrate									
D 1500	4.0	3.0	1.5	3.0	6.5	0.5	4.5	2.5	1.0
Oil Comparator	4.0	3.5	1.5	3.0	6.5	0.5	4.0	2.5	0.5
150°C (3 hr Aging), Filtrate from field device filtration									
D 1500	6.5	4.0	1.5	4.0	7.0	0.5	5.0	4.5	1.0
Oil Comparator	7.0	4.0	2.0	4.0	7.0	0.5	5.0	5.0	1.0

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*See Appendix B.

The filterability ratio test for cleanliness was performed on four of the fuels. This test measures the time to filter a given amount of contaminated fuel and then compares this to the filtering time for an equal portion of the same fuel that has been prefiltered. A value of 1 for the test indicates a perfectly clean fuel. Table 10 contains the results of this test. This procedure could very well be used in conjunction with the EOP to evaluate fuel cleanliness. However, this test requires a constant vacuum source which would eliminate the use of a hand pump. It may be possible to perform this test in a field environment with over-pressure on the input side of the filter being used to create a differential pressure across the filter, rather than using vacuum on the output side to create the differential. The low volume and low pressures required could be supplied from a small tank of compressed air or inert gas through a suitable constant pressure regulator. However, this approach has not as yet been tested.

TABLE 10. RESULTS OF FILTERABILITY RATIO TEST AND FILTRATION BY ASTM METHOD D 2276

Sample Code No.	6624	6625	6626	6630
Filterability Ratio, 1.2 μm, 500 ml samples	28.30	1.32	1.22	1.67
Particulates, D 2276 mod., 1.2 μm, mg/500 ml	13.10	1.00	1.36	15.58

To estimate the stability of the fuel, samples were heated at 150°C for 3 hours. Table 11 contains the data from this aging procedure. Included in the table are the particulate weights from both the field filtering device filtration and the D 2276 filtration. Also included in Table 11 are the results of the ASTM Method D 2274 test of the neat fuel.

TABLE 11. COMPARISON OF 150°C TEST DATA
TO DATA FROM ASTM METHOD D 2274

Sample Code No.	D 2274, Total Insolubles, mg/100 ml	150°C Test, (3.0 hr aging) Particulate Weight, mg/100 ml, 1.2 μm Field Filtering Device	D 2276
6638	0.1	0.1	0.1
6626	0.2	0.9	1.1
6746	0.3	1.4	1.1
6624	0.6	5.3	5.3
6678	1.0	3.7	4.5
6625	2.0	2.7	3.1
6630	2.1	7.8	10.0
6574	7.4	12.5	6.7

After examining the data, it was judged that although a linear relationship does not seem to exist between the 150°C test data and the D 2274 test data, a go/no-go type value for the 150°C test might be determined. It was also decided to shorten the aging period to 1.5 hours rather than maintaining the 3.0-hour time. Not only was 3 hours considered too long for a field test, but it was also thought to be more extreme than necessary.

The data generated during the evaluation of the laboratory prototype indicated that the approach to measuring the stability and cleanliness of fuel was a viable one. However, some areas where improvements were needed were identified. All of the electrical instruments in the prototype device were dependent upon 120 volts alternating current (VAC) for power. This dependence upon line current was a limiting factor to in-field use. This problem needed to be resolved to increase the usefulness of the device. Additionally, a stable power supply for both the lamp and the detector was needed. Conversion of existing circuitry or locating a lamp and detector that could be powered by one or two 12-volt batteries were possible solutions.

Color bodies from the fuel tended to adsorb onto the membrane filters. If water was present in the fuel the sample would filter more slowly through the membrane filters or possibly not filter at all. The filter holder needed to be improved so that fuel did not flow around the filters.

IV. PORTABLE FIELD FUEL QUALITY MONITOR

Based on information gained during the evaluation of the laboratory prototype field quality monitor, it was decided to construct a portable field unit. This field unit is capable of performing each of the functions performed by the laboratory prototype device, and with the exception of a heating block for stability testing, is battery powered. A more detailed test procedure for use of the fuel quality monitor is found in Appendix B.

A. Experimental

Again the flow chart pictured in Figure 1 was used as a basis for evaluation. In order to sample the fuel from its storage container, an apparatus such as the one pictured in Figure 5 was used. A vacuum was applied to the sample container by using the hand pump, and the fuel was drawn into the can through the tubing.

FIGURE 5. FUEL SAMPLING APPARATUS

To filter the fuel, the apparatus pictured in Figure 6 was used. The equipment pictured in Figures 5 and 6 as well as other equipment is packaged in a separate case as shown in Figure 7. A more detailed listing of the equipment is found in Appendix B. The electric vacuum pump, rather than the hand pump shown in Figure 5, was used for filtering in order to maintain a more uniform and sustained vacuum. The filtering apparatus pictured in Figure 6 was used in preference to the device used for filtering in the laboratory prototype. The filter holder in the newer apparatus does not allow fuel to flow around the filters as did the polycarbonate filter holder. A 500-milliliter aliquot was taken from the fuel sample and filtered through two glass fiber filters. Glass fiber filters were used because membrane filters adsorb color bodies which may alter the readings. It is important to note that the glass fiber filters used have a nominal porosity of 1.2 micrometers. These two filters (a sample filter and a control filter) are rated for amount of particulate matter present using the Contaminated Filter Measuring Device (CFMD) pictured in Figure 8. The CFMD is essentially a field-portable model of the EOP described earlier. The CFMD rates a filter by beaming light through it and measuring the amount of light that passes through. The readings for the sample and control filters were converted to %T and absorbance values and the results were compared to a calibration curve to estimate the amount of particulate matter present.

FIGURE 6. FUEL FILTERING APPARATUS

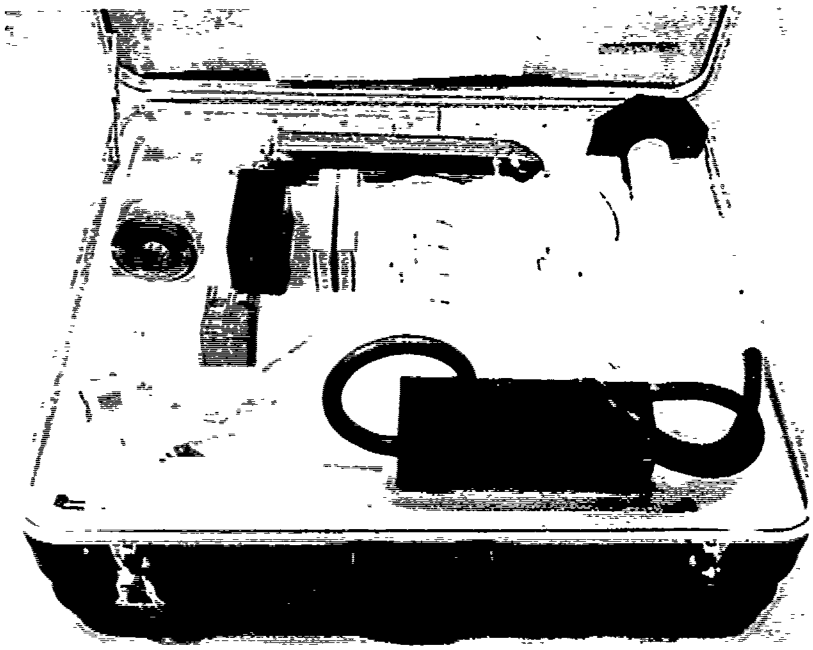

FIGURE 7. SAMPLE HANDLING AND FILTERING EQUIPMENT

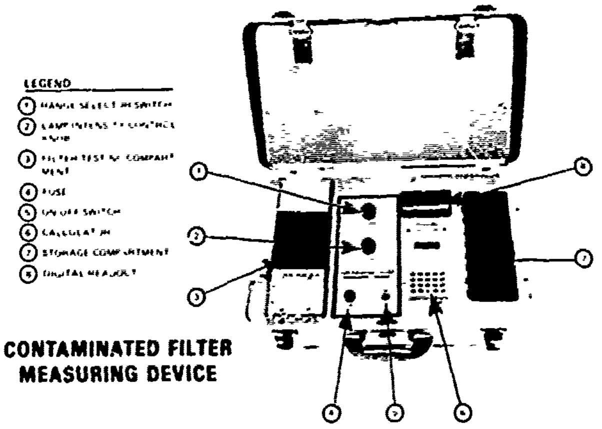

FIGURE 8. CONTAMINATED FILTER MEASURING DEVICE

The color comparator described in the preceding section was used to measure the color of the fuel. This device is small, portable, and does not require a special light source. The device gives results that are comparable to results from ASTM Method D 1500.

To age the fuel, 50-milliliter fuel samples are prefiltered, placed in test tubes, and heated to 150°C for 90 minutes in a small 115 VAC heating block as pictured in Figure 9. This aged fuel was then filtered using glass fiber filters, and the filters were rated using the CFMD.

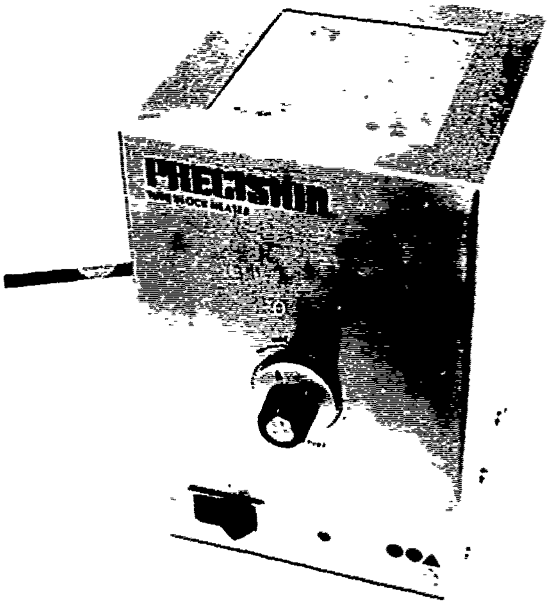

FIGURE 9. FUEL HEATING BLOCK

Figure 10 is a block diagram for the CFMD and Figure 11 is the schematic for the CFMD. To measure the amount of particulate matter present in a fuel, a sample of the fuel is filtered through sample and control filters. The sample filter is placed into the test chamber. The amount of light which passes through the filter is detected by the photocell. The output of this photocell is amplified and displayed as a voltage on a digital panel meter. Within the detection range of the photocell, this voltage output is a linear function of light intensity at the detector. The reading for the sample filter is recorded, and then the control filter is measured in a similar fashion.

The prototype device as constructed uses a photocell near the light source in a feedback loop to the lamp's power supply. This assures that the light, once set to a particular brightness level, will remain at that same brightness level as long as the main power is not shut off.

The photocell continuously monitors the lamp intensity, and if the intensity increases above the set brightness, the voltage from the supply decreases to return the brightness to the correct level. If the lamp intensity decreases below the set brightness, the supply voltage increases until the set brightness level is reached. Thus the feedback loop continuously monitors, tracks, and adjusts illumination to maintain a constant output from the light bulb. This constant level of light output is important since each of the filters must be illuminated by the same brightness level to allow accurate measurement of relative transmission percentages. While this approach works quite well, it does have the disadvantage of high power consumption in the regulation circuit. An alternative approach would use two sample chambers, one for each filter, and would compare photocell outputs simultaneously. This would decrease testing time and eliminate the need for the regulation circuit, since any light variations would affect both sample chambers by the same amount. Direct comparison of photocell output ratios could be expressed as percent transmission requiring any calculation steps. A program is currently underway to reduce electrical power consumption and simplify the CFMD through an approach using dual detectors.

The light intensity measured through the sample filter is less than that measured through the control filter due to the attenuation of light transmission by the particles on the filter. These two light intensity readings are then used to calculate %T and absorbance values as given in Table 7. Using this technique, any measured reduction in light transmission through the sample filter as compared to the control filter is caused by the light-blocking effects of the trapped particles. Calibration charts are developed by correlating measured weights of particles to measured transmission of light through the filter.

An experiment was conducted to determine whether the initial intensity of the lamp would have any effect on the measured %T of the filters. Under this experiment, various neutral density filters were measured for %T at varying degrees of lamp intensity. The results of this experiment are given in Table 12. Examination of the data shows that the initial intensity of the lamp had no significant effect on the measured %T of each of the neutral density filters. Hence, a calibration curve developed at a given intensity will in general be valid at all intensities.

A calibration curve was developed for the CFMD by filtering various fuels through two glass fiber filters and determining the %T and absorbance of the filters (see Table 7). Table 13 lists the data generated to develop the calibration curve, and Figure 11 shows the curve that was developed using the absorbance data. In addition, a calibration curve may be drawn using the %T data (Figure 12). It is preferable to use Figure 12 as the calibration curve as opposed to Figure 13 because the curve in Figure 12 is more linear than that in Figure 13, and hence data interpolation is more readily accomplished and is more precise.

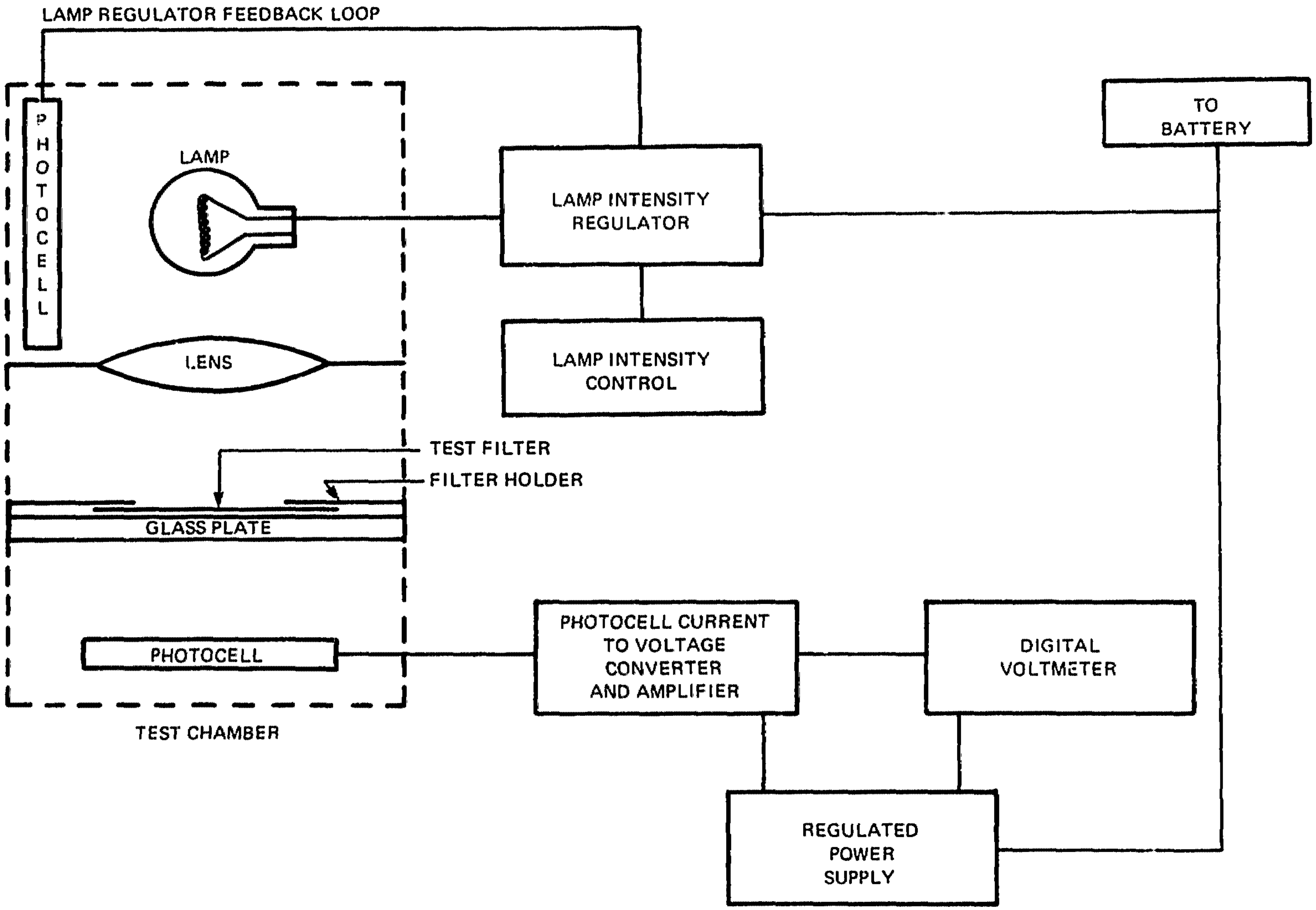

FIGURE 10. BLOCK DIAGRAM FOR CONTAMINATED FILTER MEASURING DEVICE

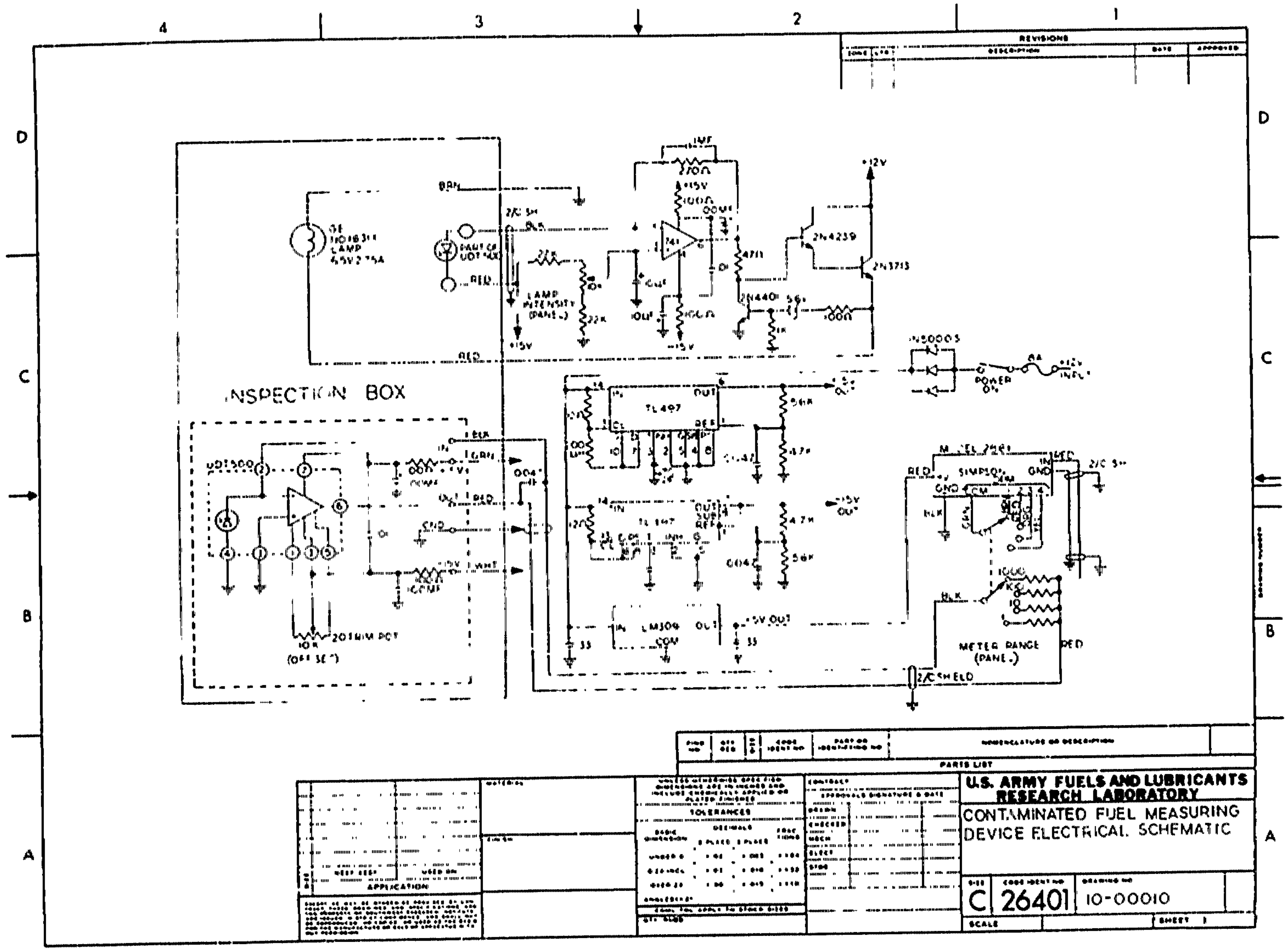

FIGURE 11. WIRING SCHEMATIC FOR CONTAMINATED FILTER MEASURING DEVICE

TABLE 12. RESULTS OF MEASURING TRANSMITTANCE THROUGH KODAK WRATTEN NO. 96 NEUTRAL DENSITY FILTERS AT VARIOUS INITIAL LIGHT INTENSITIES

Neutral Density Filter A; Density =0.60

Initial Light Intensity	Transmitted Intensity*	Calculated %T	Manufacturer's %T
4.00**	1.27	31.8+	25.0
6.00	1.90	31.7	25.0
8.00	2.51	31.4	25.0

Neutral Density Filter B; Density =0.40

Initial Light Intensity	Transmitted Intensity*	Calculated %T	Manufacturer's %T
4.00	1.85	46.3	40.0
6.00	2.77	46.2	40.0
8.00	3.68	46.0	40.0

Neutral Density Filter C; Density =0.20

Initial Light Intensity	Transmitted Intensity*	Calculated %T	Manufacturer's %T
5.00	2.87	57.1	63.0
6.50	3.72	57.2	63.0
7.91	4.52	57.4	63.0

Neutral Density Filter D; Density =0.10

Initial Light Intensity	Transmitted Intensity*	Calculated %T	Manufacturer's %T
4.00	3.10	77.5	80.0
6.00	4.64	77.3	80.0
8.00	6.16	77.0	80.0

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* The intensity of the light which passes through the filter.

** Note: Light intensity values are relative intensity values as read in the volts from the units digital voltmeter.

+ Note: Variations from manufacturer's nominal %T rating as compared to calculated %T is within manufacturing tolerance of this filter type.

TABLE 13. DATA USED TO DEVELOP A CALIBRATION CURVE FOR THE CONTAMINATED FILTER MEASURING DEVICE

Particulate Weight, mg	CFMD Readings	
Glass Fiber Filter	%T	Abs.
1.08	80.8	0.09
1.48	64.9	0.19
3.04	64.9	0.32
3.48	49.3	0.31
5.08	31.3	0.50
5.20	40.5	0.39
5.72	43.6	0.36
6.28	34.4	0.46
6.64	36.5	0.44
7.96	19.6	0.71
8.04	27.9	0.55
8.04	27.5	0.56
9.48	17.3	0.76
12.52	12.0	0.92
13.68	10.5	0.98
16.48	5.3	1.28

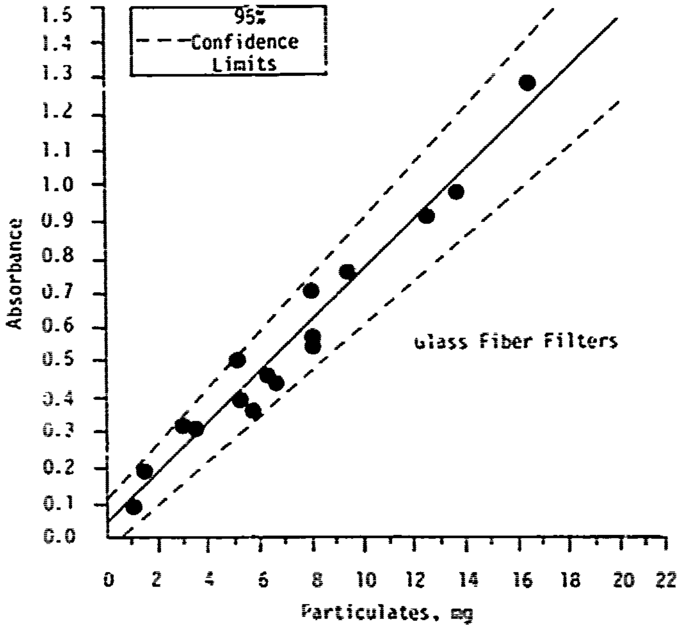

FIGURE 12. CALIBRATION CURVE FOR CONTAMINATED FILTER MEASURING DEVICE—ABSORBANCE VERSUS MG PARTICULATES

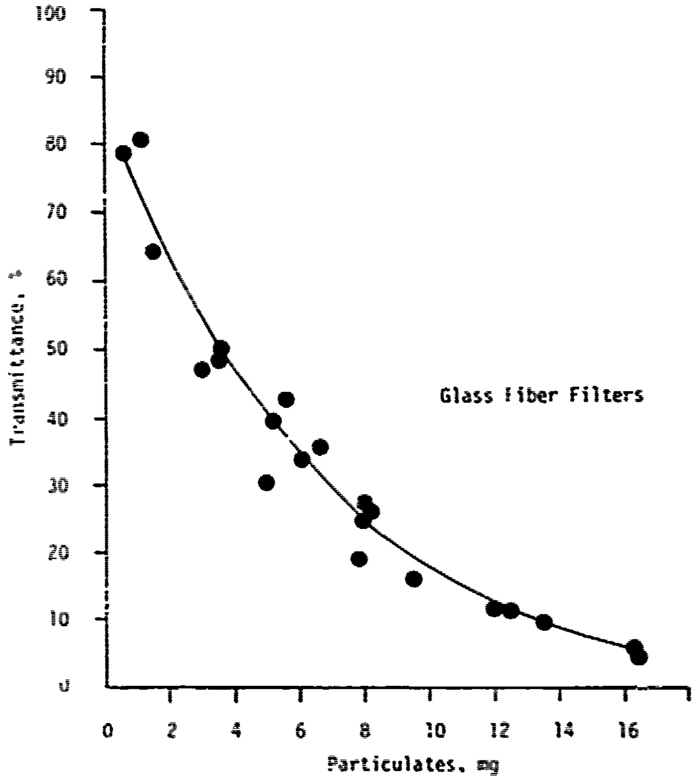

FIGURE 13. CALIBRATION CURVE FOR CONTAMINATED FILTER MEASURING DEVICE—PERCENT TRANSMITTANCE VERSUS MG PARTICULATES

B. Discussion

The portable field unit has undergone limited field testing. Sites at which field testing has been conducted include the AFLRL, Fort Hood, Texas, and the POMCUS Fully Fueled Vehicle Test in Miseau, West Germany. Table 14 is a listing of sample data from the various test sites comparing particulate values determined using the field test unit with values determined by laboratory gravimetric analysis. Note that in both cases, glass fiber filters were used. The results show reasonably good agreement between corresponding values considering that field conditions are less conducive to test accuracy.

TABLE 14. COMPARISON OF PARTICULATES DATA OBTAINED USING THE CFMD AND IN LABORATORY ANALYSIS

	Particulates mg/1000 ml, Glass Fiber Filter	
Sample	Determined in the Field Using the CFMD	Determined by Laboratory Analysis
1	0.1	1.1
2	1.0	1.1
3	1.4	1.4
4	1.5	1.8
5	2.1	2.5
6	2.4	4.4
7	2.5	5.1
8	2.7	4.2
9	3.3	1.9
10	4.1	5.0
11	4.7	4.5
12	14.9	15.6

Table 15 is a comparison of particulate weights determined using glass fiber filters and the CFMD, and particulates determined by ASTM Method D 2276, modified (1.2-micrometer membrane filters). This data shows that generally there is good agreement between the CFMD results and standard laboratory analysis. However, additional correlative data over a larger range are needed for D 2276 (1.2-micrometer) and glass fiber filtration (both gravimetric and CFMD determined.)

TABLE 15. COMPARISON OF PARTICULATE DATA GENERATED BY THE CFMD AND BY ASTM METHOD D 2276

Sample	CFMD, glass fiber, mg/1000 ml	D 2276 mod., 1.2μm, mg/1000 ml
1	1.0	1.1
2	1.4	1.4
3	1.5	1.8
4	2.1	2.5
5	2.4	4.4
6	2.7	4.2
7	2.7	5.1
8	3.2	2.0
9	4.1	5.0
10	4.6	4.5
11	5.1	7.1
12	8.4	8.8

The use of a particulates value of 4 mg/100 ml for the 150°C aging as a go/no-go value for approximating fuel stability is primarily based on previous experience which tends to indicate that a fuel with a 150°C test result of 4.0 mg/100 ml or less will, in the majority of cases, have a result of 1.5 mg/100 ml or less for ASTM Method D 2274. This guideline value can be better defined after experimentally measuring a larger statistical sampling of fuels.

It is noted that the fuel samples aged in the 150°C test are prefiltered before aging. This procedure conforms with the 150°C test proposed by ASTM. However, the prefiltration does, of course, reduce the total particulates as measured after aging and may also remove material which, if left in the fuel, would act to increase particulate formation.

Figure 14 is a plot of particulates data from the 150°C test (1.5-hr) versus total insolubles from ASTM Method D 2274. This plot is divided into areas of good, marginal, and bad as determined by the limiting values discussed in the background section of this report (Table 4).

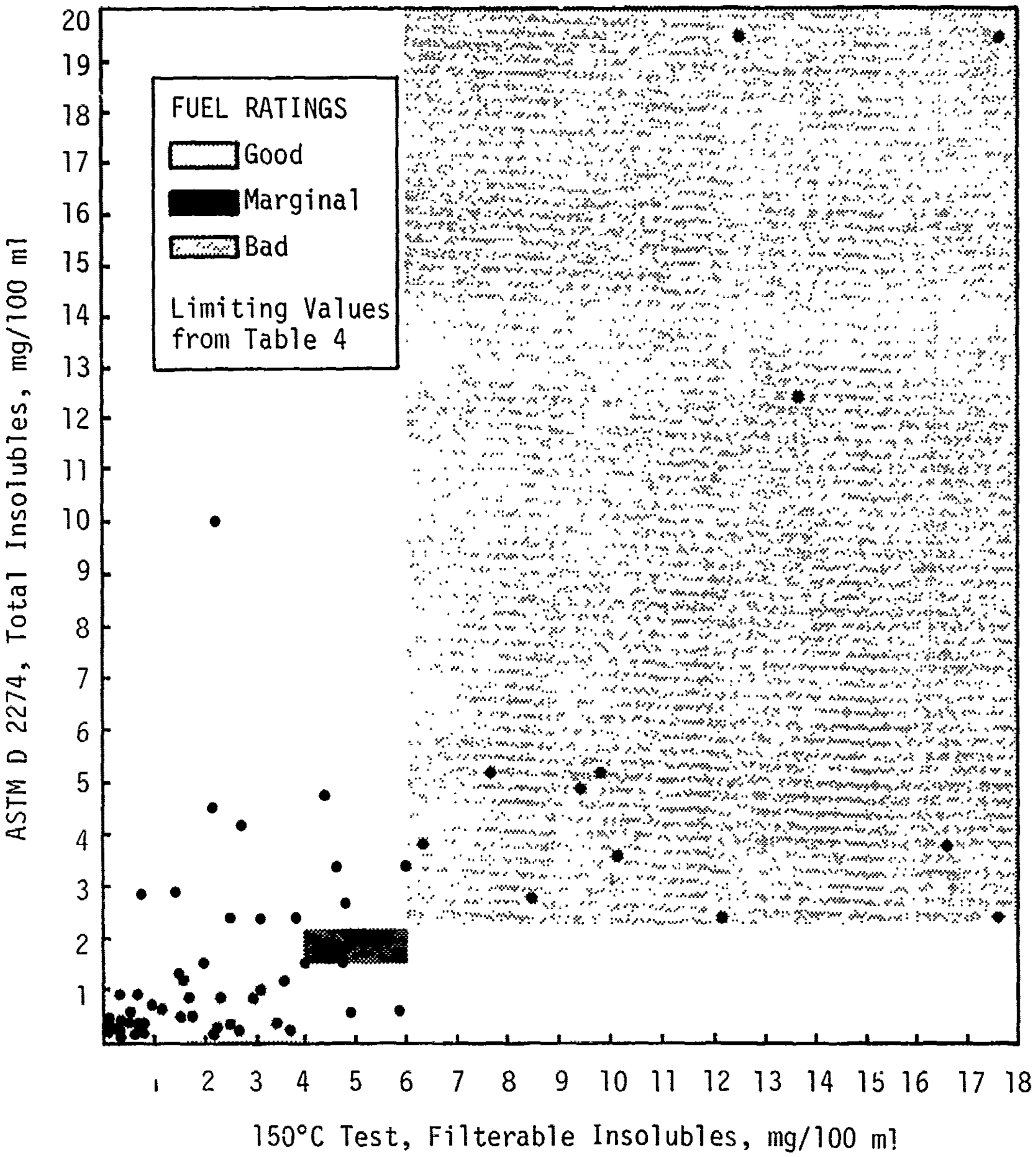

FIGURE 14. PLOT OF 150°C TEST DATA VERSUS TOTAL INSOLUBLES FROM ASTM METHOD D 2274

Table 16 is a comparison of particulates data from the 150°C test (1.5-hr) with total insolubles data from ASTM Method D 2274. These data are for samples from various test sites. With regards to measuring fuel stability, the 150°C test is better used in a trend analysis type situation. However, the data in Figure 14 and Table 16 indicate that a go/no-go particulate value of 4 mg/100 ml for the 150°C (1.5-hr) test is a reasonably good indicator of good inherent stability. In the case where the 150°C test results are close to 4.0 mg/100 ml, it may be desirable to run further laboratory tests to confirm the 150°C results and/or alternatively monitor the fuel particulate formation in storage.

TABLE 16. COMPARISON OF 150°C (1.5-HR) DATA WITH ASTM METHOD D 2274 TOTAL INSOLUBLES ON SAMPLES FROM VARIOUS TEST SITES

Test Site	Particulates, 150°C (1.5-hr) Glass Fiber*	D 2274, Total Insolubles, mg/100 ml
Ft. Hood	2.3	0.5
	9.5	3.8
Marine Storage	2.7	0.1
	5.5	0.2
	1.2	0.2
	7.3	0.2
	2.1	0.4
	4.9	0.5
	2.2	0.6
	6.0	2.9
	15.0	3.6
	8.9	4.6
	7.5	5.6
	10.0	6.1
	17.6	6.1
	8.3	8.0
	13.7	9.0
Ft. Irwin	4.7	1.5
	17.6	2.4
	3.8	2.4
	6.0	3.4
	23.9	3.8
	9.4	4.5
	9.8	5.2
	17.6	19.4
TACOM	1.5	0.2
	1.9	0.2
	2.3	0.3
	2.7	0.3
	2.5	0.3
	2.1	0.3
	1.0	0.4
	1.8	0.4
	1.1	0.4
	1.7	0.5
	1.4	0.5
	7.3	0.5
	0.1	1.0
	0.1	1.0

*Particulates determined by ASTM D 2276, modified to use glass fiber filters.

C. Comparison of NAVSHIPS Contaminated Fuel Detector to Field Fuel Quality Monitor

A device used to measure the cleanliness of fuel is commercially available(3) and currently in use by the United States Navy. This device is called the NAVSHIPS Contaminated Fuel Detector (NSN: 2H6630-706-2302). At least three models have been or are being used: models EMSE 363, 373, and 383. Models 363 and 373 have been used for years, whereas model 383 has only recently come into regular use.

The NAVSHIPS device, such as the model 363 pictured in Figure 15, measures fuel cleanliness in much the same manner as does the field device described above; however, the NAVSHIPS device was developed to measure the cleanliness of aircraft fuels. When the device is used with diesel fuel, some problems arise. Diesel fuel tends to be darker in color and more contaminated than aircraft fuels, and therefore the calibration chart provided with the instrument is not satisfactory for use with diesel fuels because the linear range is too small.

During derivation of a new calibration curve for the NAVSHIPS device, examination of the wiring diagram for models 363 and 373 (Figure 16) showed that the photocell is directly coupled to the milliammeter. At low light levels, such as encountered with very contaminated filters and filters stained with dark color bodies, the impedance of the meter and the energy required to deflect the meter's pointer effectively load the photocell output. This loading effect produces meter readings which are lower than they should be. The magnitude of the error decreases as the intensity of the light reaching the photocell increases.

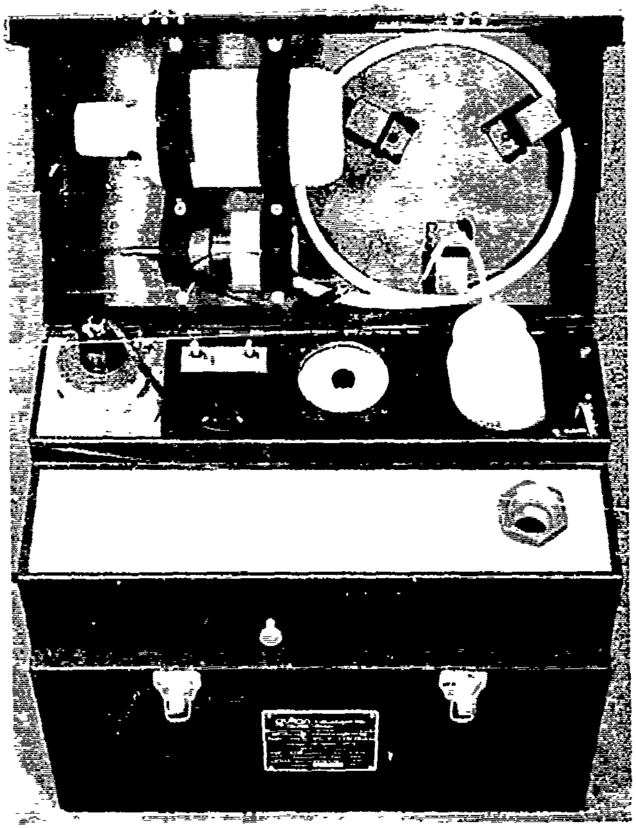

FIGURE 15. NAVSHIPS CONTAMINATED FUEL DETECTOR

To correct for this discrepancy in the meter readings, a Meter Correction Chart (see Figure 17) was developed. The correction chart was generated by comparing the %T through various neutral density filters at several light intensities in both the NAVSHIPS device and the CFMD. A calibration curve (Figure 18) was developed in the same manner as the calibration chart for the CFMD. The circuitry in the CFMD was designed to be linear at all light levels and does not require the use of a meter correction chart. The light measuring circuit of the newer NAVSHIPS Model EMSE-383 has been modified to include an amplifier stage between the photocell and the meter. This amplifier serves as a buffer to prevent the loading effect. In addition, the newer model is also fitted with a digital readout to make readings simpler and more precise.

Using the correction chart, an operator can correct meter readings to measure more accurately the actual transmittance through a filter. A model 373 has been used with some success in a joint U.S. Marine Corps (USMC)/U.S. Army Mobility Equipment Research and Development Command (MERADCOM) fueled vehicle storage test.(6) The purpose of the test is to assess the feasibility of storing fueled vehicles on board ships. The test vehicles are partially filled with additive-treated fuel. The NAVSHIPS device, which was calibrated at AFLRL, is used to give on-site assessment of the fuel quality. Table 17 is a comparison of particulates data generated on-site using the NAVSHIPS device and data from laboratory analysis of samples shipped to AFLRL. Examination of the data shows good agreement in most cases.

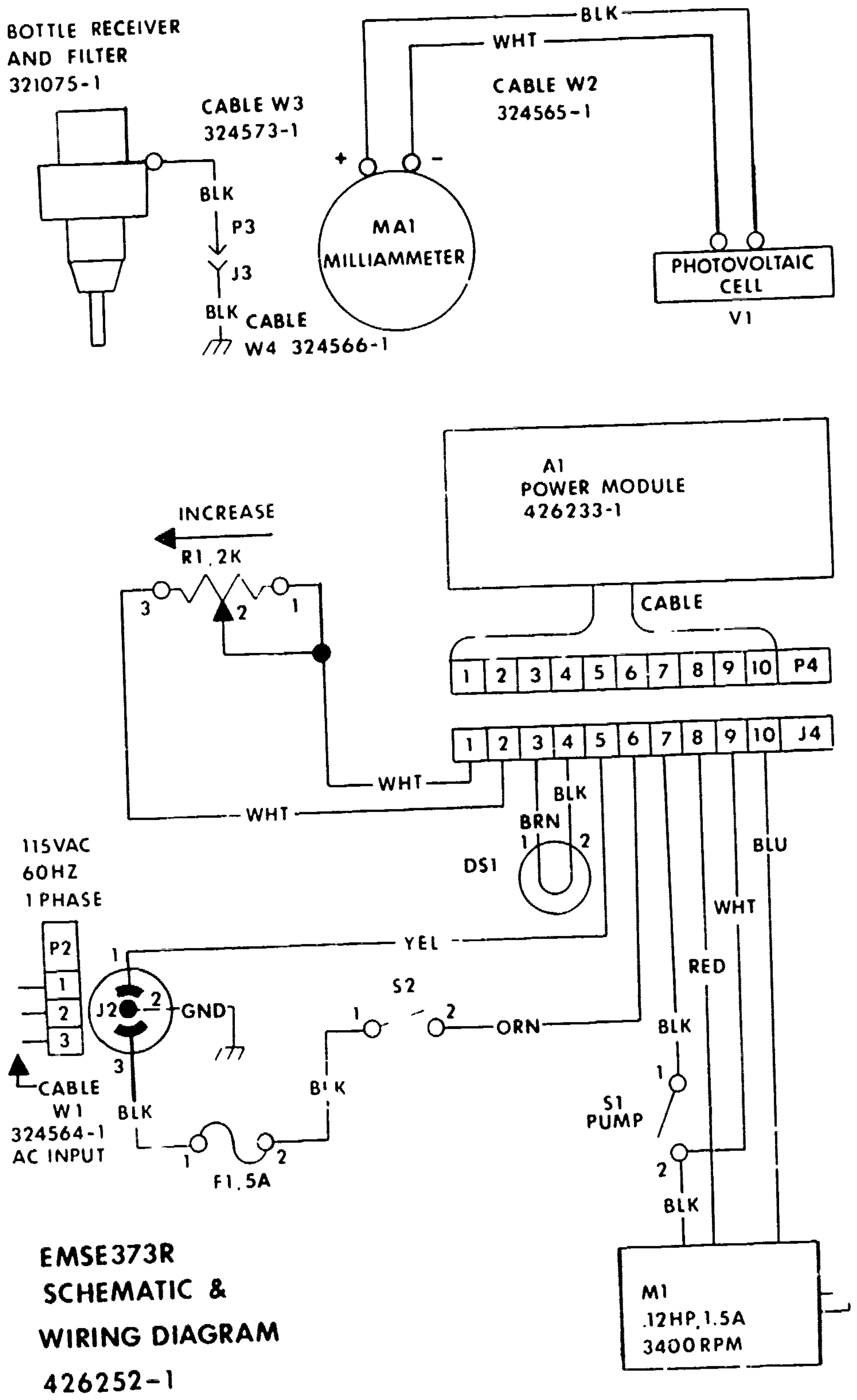

FIGURE 16. SCHEMATIC AND WIRING DIAGRAM FOR NAVSHIPS EMSE 363 AND EMSE 373

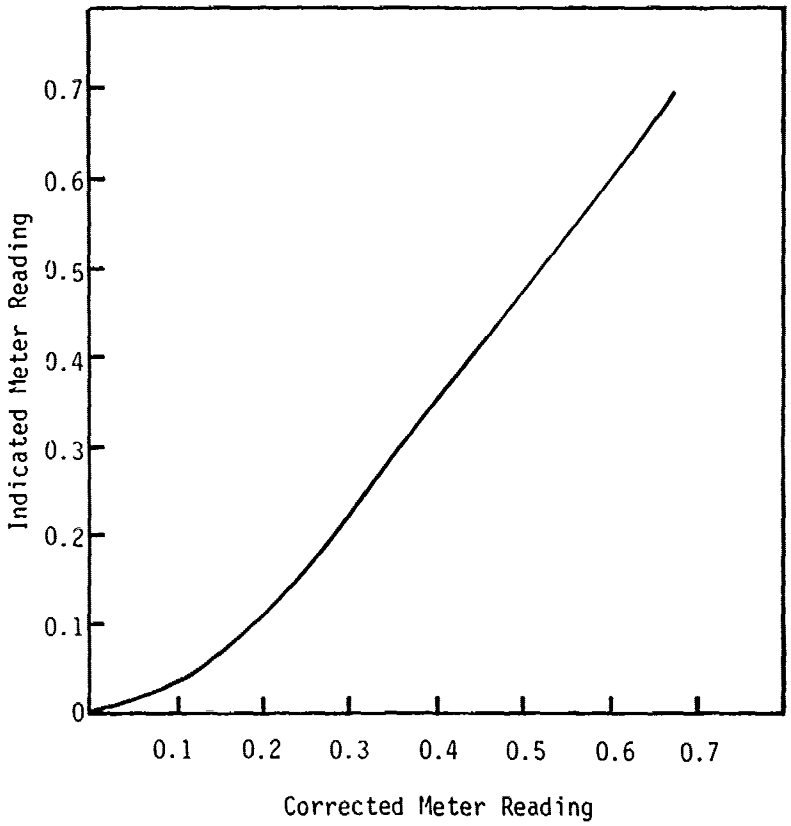

FIGURE 17. METER CORRECTION CHART FOR NAVSHIPS CONTAMINATED FUEL DETECTOR

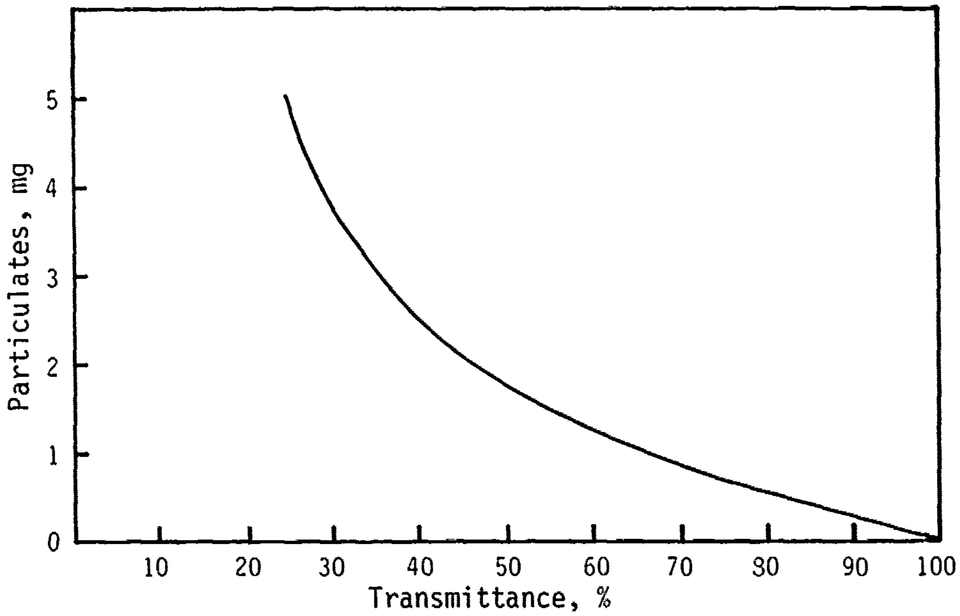

FIGURE 18. CALIBRATION CURVE FOR NAVSHIPS CONTAMINATED FUEL DETECTOR

TABLE 17. COMPARISON OF PARTICULATES DATA FROM THE USMC/MERADCOM FUELS TEST

Vehicle Code No.	D 2276, 1.2-μm membrane, mg/1000 ml (At AFLRL)	Particulates CFMD Glass Fiber, mg/1000 ml	Marine Field Test, Glass Fiber Filter, mg/1000 ml (At the Test Site)
B-7	0.3	0.5	0.3
A-10	1.2	0.8	0.5
A-7	1.5	1.6	0.8
B-2	10.3	10.0	11.4
B-3	16.8	17.0	16.4
A-2	17.5	18.2	14.4
B-5	26.4	25.4	off scale
A-4	40.3	off scale	off scale
A-1	56.6	off scale	off scale

When comparing the NAVSHIPS device to the AFLRL unit, it must be considered that each was developed to perform a slightly different function. The NAVSHIPS device was constructed to measure the cleanliness of relatively clean aviation fuel whereas the AFLRL unit was developed to test a wider range of cleanliness levels with an emphasis on diesel fuels. The NAVSHIPS device requires 115 VAC for power while the AFLRL unit was designed to be used in the field and requires 12 to 24 volts direct current (VDC). The NAVSHIPS device is more compact; however, it contains no equipment for aging fuel. With the exception of the model 388, the NAVSHIPS device requires a meter correction chart for use with diesel fuel.

V. CONCLUSIONS, RECOMMENDATIONS, AND FUTURE PLANS

A portable unit to measure the quality of distillate fuel in the field was developed.

The feasibility of using light transmittance through a filter to measure the amount of contaminant present on a filter and thereby measure the cleanliness of a distillate fuel sample was shown. Light transmittance and gravimetric analysis were correlated for particulates on test filters. It was determined that initial lamp intensity of the Contaminated Filter Measuring Device (CFMD) had essentially no effect on the measured transmittance through the filter.

Cellulose acetate/cellulose nitrate-type membrane filters were judged unsuitable for use with the light transmittance method of quantitating particulates because polar color bodies in the fuel adsorbed onto the filter media, causing false reading. Glass fiber filters of the type used in a standardized accelerated stability test (ASTM Method D 2274) were found to be more suitable.

A screw-together, cartridge-type filter holder was found unsuitable for in-the-field filtering of diesel fuel because it allowed fuel to flow around the filter. Therefore, a clamp-together filter holder of the type used in ASTM Method D 2276 was used.

A commercially available oil comparator was used to test fuel color and gave results equivalent to ASTM Method D 1500.

A method of estimating fuel stability was demonstrated. A sample of the fuel is heated to 150°C for 90 minutes, the fuel is then filtered, and the amount of particulate matter formed is measured by the light transmittance technique. The test results are compared to other accelerated test procedures and pre-established limiting values to rate the fuel as good, marginal, or bad.

The commercially available NAVSHIPS unit may be used to measure a limited range of particulate contamination, but only where a 110 VAC power source is available. The NAVSHIPS unit has no provision for aging fuel to estimate fuel stability.

A number of recommendations were identified:

- The CFMD should be reduced in size to allow greater ease of handling, storage, and transportation.
- The lamp regulator feedback loop consumes much of the energy used by the CFMD. The dual chamber/simultaneous comparison configuration using dual light detectors should be investigated as a means of reducing this waste of power as well as simplifying and shortening the actual test procedure.
- A safety hazard currently exists with the method used to heat the fuel to 150°C for stability testing. This matter should be studied further, and ways to reduce the hazard should be identified and implemented. The heating block used to age the fuel is also quite heavy. Possible methods of reducing the size and weight of the heater should be investigated.
- The prototype AFLRL field unit has undergone limited testing, but should be subjected to a more thorough user acceptance testing program.

A field test program to demonstrate the Diesel Fuel Quality Monitor utility/effectiveness has been planned for initiation in late 1981.

VI. REFERENCES

1. Stavinoha, L.L., Westbrook, S.R., and LePera, M.E., "Army Experience and Requirements For Stability and Cleanliness of Diesel Fuel," AFLRL Report No. 128, U.S. Army Fuels and Lubricants Research Laboratory, Southwest Research Institute, San Antonio, TX, Government Accession No. AD A088008, July 1980.

2. Commercially available from Gulton Industries Inc., Hawthorne, CA and Telectro-Mek Inc., Fort Wayne, IN.

3. Memorandum from J.H. Frazar, U.S. Army Fuels and Lubricants Research Laboratory, to file 10-5070-140, with the subject, "Field Test Kit for Diesel Fuel Contamination," 5 March 1979.

4. Described in "Operation and Maintenance Manual for Test Kit, Fuel Contamination," prepared for Headquarters U.S. Army Troop Support Command by Potomac Research, Incorporated, December 1974. Note: Not an official DA Publication and not available through AG publication channels.

5. Stavinoha, L.L. and Westbrook, S.R., "Accelerated Stability Test Techniques for Middle Distillate Fuels," presented at the American Society for Testing and Materials (ASTM) Symposium on Distillate Fuel Stability and Cleanliness, Chicago, IL, Special Technical Publication, No. 751, 24 June 1980.

6. Correspondence to U.S. Army Mobility Equipment Research and Development Command, DRDME-GL, from S.R. Westbrook, U.S. Army Fuels and Lubricants Research Laboratory, with the subject, "Stability Additive Package Evaluation in Partially Fueled Vehicles on Board USMC Ships at Diego Garcia; Laboratory Results of February 1981 and March 1981 Samples," 2 June 1981.

APPENDIX A

EXPERIMENTAL TEST PROCEDURES FOR DISTILLATE FUELS

APPENDIX A
EXPERIMENTAL TEST PROCEDURES FOR DISTILLATE FUELS

The following experimental methods are used to test distillate fuels:

1. Color: ASTM D 1500-64

2. Steam Jet Gum: ASTM D 381-70 (Modified)
*Modification:*The residue is dried an extra 0.5 hr in the gum block if it still appears wet after the first 0.5 hr.

3. Particulate Contamination: ASTM D 2276-7 (Modified)
Modification:
- 47-mm diameter filters are used instead of 37-mm diameter filters.
- 1.2-micrometer pore size filters are used instead of 0.8-micrometer pore size filters.
- *n*-heptane is used as the solvent.
- The sample size may range from 100 to 1000 ml.

4. Accelerated Stability: ASTM D 2274-74.

5. NACE Standard TM-01-72 Steel Corrosion: Antirust properties of petroleum products pipeline cargoes test method.

6. 43°C Storage Test: This test method is used for studying the storage stability of distillate fuels under vented 43°C storage conditions.

The bottles used were made of borosilicate glass (Curtis Matheson Scientific, Inc., Catalog No. 037-59 (1977), are rectangular (the cross section is 6.35 cm x 9.53 cm), and are 27.31 cm in overall height. The volume of each bottle is 1 liter. The bottles have a tool-finished neck for increased strength, with an opening that takes a No. 6 rubber stopper. A 10.2-cm square, thin sheet of Teflon was wired securely around the neck to cover the opening. A 0.64-cm diameter hole was punched into the sheet for insertion of a bent piece of glass tubing to act as a vent.

The storage bottles were cleaned with alkaline laboratory cleaner and then rinsed with water. Each bottle was filled approximately half full with chromic acid cleaning solution and rolled in such a way that all of the inner surface came in contact with the acid solution. The bottle was allowed to stand for at least 1 hr before it was rinsed four times with deionized water. The bottles were allowed to drain and then dry overnight in an oven at 150°C.

The fuels to be tested were prefiltered through a 1.2-micrometer membrane filter (Millipore Corp., Bedford, MA, Catalog No. RA-WP-047-00). Then four samples (650 ml per sample) of each fuel under study were placed in the bottles and properly labeled. The perforated caps previously described were placed on the bottles.

One bottle from each series was retained for analysis at zero weeks storage. The control samples were stored in the dark under nitrogen at -1° to 4°C until analyzed. The remaining bottles were placed in the 43°C oven. One bottle of each fuel was removed after 12, 16, and 32 weeks of storage. The samples were carefully inspected for the appearance of visual sediment and were analyzed for color by ASTM Method D 1500. Filterability ratio (modified) and steam jet gum were also determined. Then 100 ml of each sample were filtered, using a modified D 2276 method, through 1.2-micrometer membrane filter (as described above). The filter was dried and weighed. The resulting particulates were reported as mg/100 ml.

7. 80°C Storage Test: In this test, ten 120-ml volume sample bottles were used for each fuel. The caps of these bottles were fitted with a Teflon liner, and each liner was pierced with a 0.65 cm diameter hole. A bent glass tube was placed in the hole to act as a vent. Each bottle contained 105 ml of sample.

One control sample of each fuel tested was set aside under nitrogen and in the dark. The other nine samples of each fuel were placed in a force-draft oven at 80°C. Of these samples, three of each fuel were removed from the oven after 24 hr, another three samples after 72 hr, and the final sample of fuel after 168 hr. The samples were tested for color (ASTM D 1500), light absorbance, steam jet gum (D 381), visual sediment, and particulates (D 2276, modified, 1.2-micrometer pore size filter).

8. 150°C Accelerated Fuel Oil Stability Test: In this test, three 55-ml samples of a fuel were measured into 2.5 cm x 20.0 cm culture tubes with screw caps. These tubes were Kimax Brand glass from Curtis Matheson Scientific, Inc., Catalog No. 225-870 (1977). The tubes containing the samples were submerged to the necks in a 150° ± 1°C oil bath. The caps on the tubes were loosened slightly to allow for air expansion in the tube. The samples remained in the batch for 90 minutes.

Two of the samples were combined and analyzed for color (D 1500), light absorbance, and particulates (D 2276, modified, 1.2-micrometer pore size filter) while the third was analyzed for jet gum (D 381).

9. ASTM D 873-74 Oxidation Stability of Aviation Fuels (modified), often referred to as the potential residue method, measures the tendency of fuels to form gum and deposits under accelerated aging conditions. The modifications made to this test and used in these studies are as follows:

- The gum solvent was replaced with *n*-heptane.
- The sintered glass crucible was replaced with a 1.2-micrometer filter using double filter tare procedure as in ASTM D 2276.
- Items A (particulate), B (insoluble gum), and C (soluble gum) were all reported as milligrams per 100 ml of sample (mg/100 ml).
- The color (D 1500) was reported for the treated sample.
- The visible sediment was reported.
- The treated sample was examined for "sour" or "oxidized linseed oil" odor and reported.

10. Light Absorbance; Spectrophotometric Analysis of Fuels: Light absorbance was measured on a UV-Visible Spectrophotometer.

The light absorbance was recorded over the wavelength scanning range of 650 to 400 nanometers (nm) at a scanning speed setting of 2 nm per second, and the light absorbance values were reported at 650, 575, 540, and 500 nm.

The instrument was "zeroed" without cells in the light path. Deionized water was placed in both cells and scanned over the 650- to 400-nm range to determine the cleanliness of the cells and any possible deviation of the baseline. The cells used were 1-cm corex cells which had been cleaned with solvents, detergents, and/or cleaning solution as required to maintain a zero baseline (0.005 absorbance units). The deionized water was removed from the sample cell, and the cell was rinsed with acetone and dried with clean air. The fuel to be examined was placed in the sample cell, and the scan was repeated. For each fuel examined, the cell was cleaned as noted previously. Periodically, the zero and baseline scan were checked with deionized water.

11. Method for Testing Fuel Filterability

•Discussion: In this method, 500-ml samples of prefiltered and neat test fuel were filtered through a 1.2-micrometer filter disc. Fuel filterability was evaluated by the ratio of the filtering time for the neat test fuel to the filtering time for the prefiltered fuel.

Equipment	Suggested Supplier
a. 1-092-10V1, Air Pump	Fisher Scientific
b. 1000-ml Filter Flask	Fisher Scientific
c. 500-ml graduate cylinder	Fisher Scientific
d. Stop watch	Fisher Scientific
e. XX10-047-00 Pyrex Filter Holder	Millipore Corporation
f. 1.2-micrometer Filter Disc No. RAWP-047-00	Millipore Corporation
g. Petri-Dishes PD-10-047-00	Millipore Corporation

APPENDIX B

PROCEDURE FOR USING THE FUEL QUALITY MONITOR AND PARTS LIST

INTRODUCTION

The monitor evaluates the cleanliness of the fuel by first filtering the fuel and then determining the amount of particulates on the filters using a contaminated filter measuring device. The filters are rated by measuring the amount of light which passes through the filters to estimate the amount of particulates based on a calibration chart. A measure of the stability of the fuel is obtained by aging a sample of the fuel at 150°C for 1.5 hours and determining the amount of sediment formed. Based on preliminary correlations of 150°C test data with ASTM D 2274 accelerated stability data, "go/no-go" values of <4, 4-6, and >6 mg/100 ml have been suggested for rating a fuel's stability as good, marginal, and bad, respectively.

OPERATING PROCEDURE

1. General Instructions.

WARNING!

Handle the equipment, components materials and fuels with utmost care. Dangers and hazards inherent in handling highly flammable liquid fuels require constant observance of and strict adherence to rules governing safety.

1.1 Be thoroughly familiar with the contents of the monitor and the uses and functions of each component before performing any test. When handling fuels, pay particular attention to the following safety precautions:

- Provide adequate ventilation.
- Permit no smoking or open flames in the testing area.
- Keep containers capped when filled with fuel.
- Handle liquids carefully to avoid spills or splashes.
- Immediately wipe up any liquid that has spilled and discard wiping material in a metal container.

1.2 Always have a chemical fire extinguisher in operating condition nearby, and know how to use it.

1.3 The following additional items are required for operation and maintenance of the monitor:

- 12 VDC power supply for electric pump.
- 12-24 VDC power supply for (CFMD); the CFMD requires at least 3.0 amps of current.
- Clean rags and detergent for general cleanup of equipment.
- Clean, metal, sample cans (preferably one-gallon size).

2. Sampling

2.1 Remove the following items from the case:

- Vacuum Pump - either hand pump or electric pump.
- One 500 ml flask and flask holder.
- Flask vacuum cap.
- Sufficient tubing to reach the appropriate sampling level in the fuel storage container.

2.2 Assemble the fuel sampling apparatus as pictured in Figure 5 of this report.

2.3 Drop the end of the sampling tube into the fuel.

2.3.1 Samples of fuel can be taken from a fuel tank or from a bulk storage source.

2.3.2 The precautions required to assure a representative sample depend on the type of product being sampled, the source from which it is drawn, and the sampling procedure employed. A procedure which is suitable for the sampling conditions should be used. (Chapter 5 of MIL-HDBK-200E gives detailed instructions for sampling.)

2.3.3 Improperly taken samples can completely invalidate a test. Only responsible and trained personnel should be assigned to obtain sample products. The importance of obtaining representative samples cannot be overstressed because analysis will not give reliable data on a product if the sample is not representative.

2.3.4 This manual will in no way alter any assigned responsibility of various activities for submitting special samples to a designated laboratory.

2.3.5 Do not take samples through storage tank cleanout lines, manifolds, water draw-offs, bleeder valves. hose nozzles, etc., as such samples will not be representative of the product in the tank. When it is necessary to sample service station tanks, and access to such tanks cannot be gained through a manhole or sampling hatch, the tanks may be sampled through the servicing hose after first discharging from the hose a volume of the product estimated at two times the capacity of the piping system.

2.3.6 It may be necessary to weight the end of the sampling tube for "deep" samples.

2.4 Apply a vacuum to the system, using either the electric pump or the hand pump, to draw the fuel into the can.

2.5 Identify each sample container immediately after sampling by securely attaching a sample tag. Information on the tag should include the location of the activity at which the sample is taken, name of person taking the sample, grade of material, quantity represented, specification of material when known, storage tank number and location, date sample was taken, type of sample, and reason for sample.

3. Filtering the Fuel Sample

3.1 Remove the following equipment from the case:

- Vacuum pump—either electric or hand pump
- 500-ml flask, flask cap, and flask holder
- Filter support, filter funnel, and clamp
- 250-ml graduated cylinder.

3.2 Push the filter support into the hole in the flask cap, place cap on flask, and attach the vacuum hose to the cap.

3.3 Using the forceps, remove two of the glass fiber filters (Note: be certain the "rough" side of the filters is up) and place them onto the filter support.

3.4 Place the filter funnel over the filters and clamp it in place.

3.5 Using the graduated cylinder measure out 250 ml of the fuel to be tested, apply vacuum to the flask, and pour the fuel into the filter funnel.

3.6 When all 250 ml of the fuel has been filtered, stop the vacuum and remove the cap from the flask.

3.7 Pour the filtered fuel into a clean container for use in testing the stability of the fuel.

3.8 Replace the cap and repeat steps 3.5—3.7. Do not separate the filter funnel from the filter support.

3.9 The filters are now ready to be tested, using the CFMD for the amount of particulates present.

4. Measuring Particulates Using the Contaminated Filter Measuring Device (CFMD)

4.1 Open case and attach the power cord to the appropriate power supply. Note that the instrument requires from 12-24 VDC and at least 2.8 amps current.

4.2 Raise the test chamber to the upright position and lock it in place.

4.3 Set the lamp intensity somewhere between 5 and 7. Set the meter range switch to 100.

4.4 Turn the power switch to the ON position and allow the instrument to warm up for ten minutes.

4.5 Open the door to the test chamber, raise the cover flap, and using forceps, place the sample filter (top filter) from the filtration procedure into the test chamber. Close the test chamber door and record the reading from the digital panel meter. Record this reading as R_s.

4.6 Repeat step 4.5 using the control filter (bottom filter). Record this reading as R_c.

4.7 Calculate the Percent Transmittance (%T) of the sample filter as follows:

$$R_s/R_c \text{ X } 100 = \%T$$

4.8 Locate the calculated %T on the calibration curve and determine the amount of particulates on the filter.

4.9 When all testing with the CFMD is complete, turn the power switch to OFF, disconnect the power lines, wipe out the test chamber with a clean, dry rag, and close the instrument.

5. Determining the Color of the Fuel

5.1 Color should be determined on the unfiltered fuel.

5.2 Open the front cover of the Comparator housing, and place the center of the Color Disc on the ring with the number plates facing the operator. Close the cover. As the Disc is rotated, the number plates representing ASTM Color Numbers can be read through the upper opening on the right-hand side of the front cover. The alignment is such that only one figure is visible when a glass Color Standard is completely in the field of view.

5.3 The observation fields for colorimetric comparison can be seen through the two central openings. Through the prism attachment, they are seen as half-fields. The right-hand field is produced by the test sample in a tube which is placed in the opening at the right of the instrument, while the left-hand field is formed by one of the glass Color Standards.

5.4 Fill a tube with the test sample to a height convenient for full exposure in the observation field and place it in the right-hand opening of the apparatus. Make the color comparison by revolving the Color Disc so that one Standard after another is brought into the observation field. When using the Comparator with the prism attachment, view the color fields with the eye in line with the center of the fields. The half-fields should be free of shadow effect which can be caused by off-center or oblique observation. For normal eyesight, the preferred viewing distance is approximately 10 inches.

Caution: Do not make the mistake of placing the eye close to the Comparator housing. Do not prolong the observation for more than 10 to 15 seconds. For very accurate readings, let the eyes rest between such intervals, preferably by viewing a gray or green surface.

5.5 When a color match is obtained between the test sample and one of the glass Standards, the figure seen in the upper opening in the front cover gives the direct reading. If the color of the test solution is intermediate between those of two glass Standards, the result to be reported will be intermediate between their corresponding values and may be estimated by interpolation.

5.6 While making readings, the opal glass plate at the back of the Comparator shall face directly toward the light, and care should be taken that pronounced shadows do not cause uneven illumination of the Comparator fields. Northern exposure provides the best daylight for colorimetric determination, but any indirect light form outdoors usually will be satisfactory. Direct sunlight should always be avoided, even in the early morning or late afternoon.

5.7 General Remarks: D 1500-64 has replaced the former ASTM Method D 155, Test for Color of Lubricating Oil and Petroleum by Means of ASTM Union Colorimeter. Method D 155 was withdrawn as an ASTM Tentative on July 1, 1960. Method D 1500 is better than the former Method D 155 in three respects:

1. The glass Standards are specified in fundamental terms.
2. The differences in chromaticity between successive glass Standards are uniform throughout the scale.
3. The lighter colored Standards more nearly match the color of petroleum products.

D 1524-64T is a Tentative Method for Visual Examination of Used Electrical Insulating Oils of Petroleum Origin in the field.

6. Measuring for Stability

6.1 Summary of Method: A measured volume of distillate fuel in an open tube is aged 90 minutes at 150°C (302°F) with air exposure. After aging and cooling, fuel is filtered and the amount of insoluble residue formed is estimated.

6.2 Apparatus: Aging tubes, glass fiber filter paper, thermometer, vacuum pump, flask, and flask holder, flask vacuum cap, filter support, filter funnel, clamp, graduated cylinder, and heating block.

WARNING! There are exposed hot surfaces on the heating apparatus which can cause severe burns. Avoid contact with exposed skin by use of protective equipment as required. The oil sample vapors must be properly vented to prevent their entering the work atmosphere. The block should be located in an open area free of sparks and open flame.

6.3 Using the graduated cylinder measure out 50 ml of the filtered fuel from the cleanliness testing procedure (paragraph 3.7 above).

6.4 Adjust the heating block to 150° ± 2°C (302° ± 5°F).

WARNING! There are exposed hot surfaces on the apparatus which can cause severe burns. Avoid contact with exposed skin by use of protective equipment as required.

6.5 Pour the 50-ml sample into one of the aging tubes.

6.6 Place the uncapped sample tube in the heating block for 90 minutes ± 3 minutes. Samples should be shielded from direct light during aging (Note 3).

WARNING! Fuels will be heated above their flash points. Fuels with initial boiling points below that of No. 2 fuel (kerosene, Jet A) may boil under the conditions of the test. Fuel samples which may contain gasoline or other volatile materials may vaporize excessively and should not be tested. The block must be located in a ventilated area (hood) and kept away from sparks and open flame.

6.7 Remove the sample from the block and allow to cool gradually to 21° to 26°C (70° to 80°F) over a period of 1.5 to 4 hours in a location shielded from light. Do not allow the samples to cool more than 4 hours.

CAUTION! The hot samples can cause severe burns. Use protective equipment.

6.8 Prepare the filtration assembly with two new filters and filter the fuel sample.

Note - If the deposit on the filter pad is not evenly distributed, the pad should be rejected and the test re-run after cleaning the filtration apparatus.

6.9 Determine the amount of particulate present on the filters as described in section 4 above.

6.10 When heating block has cooled to room temperature, clean the outside of the block only with a warm detergent solution. Also clean the aging tubes in a warm detergent solution.

LIST OF MANUFACTURERS AND SUPPLIERS

Code	Name And Address
001	General Electric Company Nela Park Cleveland, OH 44112
002	Bussmann Mfg. Div. McGraw-Edison Co. 500 EarthCity Plaza Earth City, MO
003	Eastman Kodak Co. Rochester, NY 14650
004	Curtis Matheson Scientific, Inc. P.O. Box 1546 Houston, TX 77001
005	Millipore Corporation Bedford, MA 01730
006	Neptune Products, Inc. P.O. Box 542 353 E. Blackwell St. Dover, NJ 07801
007	Spectrum Medical Ind., Inc. 48 Middle Village Station Queens, NY 11379
008	Hellige, Inc. 877 Stewart Ave. Garden City, NY
009	American Scientific Products McGaw Park, Il 60085

PARTS LIST

Part No.	Description	Manufacturer/ Supplier	Qty Inc In Unit
AL-001	Contaminated Filter Measuring Device	N/A	1 ea
GE 1631X	Lamp For CFMD	001	2 ea
MDA 8	Fuse, 8 Amp (CFMD)	002	4 ea
149 4954	Filter, Neutral Density	003	2 ea
227-348	Thermometer, 20°-500°F Range	004	1 ea
XX10 047 00	Analytical Filter Holder	005	1 ea
094-219	Filter Discs, Glass Microfiber, Reeve Angel 934-AH	004	1 Pk (100)
XX6200006	Forceps, MF Filter	005	1 ea
349-597	Pump, Vacuum, Hand Operated	004	1 ea
232-413	Tongs, Crucible	004	1 ea
279-489	Tubes, Culture, 20x150 mm	004	10 ea
4 K	Pump with 12 volt D.C. Ball Bearing Motor	006	1 ea
146323	Filtration Cap Assembly	007	2 ea
146706	Flask, 500 ml, Polypropylene	007	2 ea
146329	Stand, Vacuum Manifold	007	1 ea
607-A33	Oil Comparator	008	1 ea
600-PO	Oil Magnifying Prism Attachment	008	1 ea
607-T33	Tubes (Drawn) 33mm Viewing Depth	008	1 ea
620C-53	Color Disc	008	1 ea
620C-54	Color Disc	008	1 ea
607-0C	Directions	008	1 ea
H2090	Heating Block	009	1 ea
H2074-1	Tube Block, 8 Holes	009	1 ea
AL-002	Safety Glasses	N/A	1 Pr
AL-003	Tubing, Tygon	N/A	25 Ft

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    DRCDE-SS 1
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    DRSTA-RG (MR HAMPARIAN) 1
    DRSTA-NS (DR PETRICK) 1
    DRSTA-G 1
    DRSTA-M 1
    DRSTA-GBP (MR MCCARTNEY) 1
WARREN MI 48090

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US ARMY MATERIEL SYSTEMS
ANALYSIS AGENCY
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    DRXSY-S 1
    DRXSY-L 1
ABERDEEN PROVING GROUND MD 21005

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    AFZT-DI-M 1
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WARREN MI 48090

MICHIGAN ARMY MISSILE PLANT
PROG MGR, FIGHTING VEHICLE SYS
ATTN DRCPM-FVS-SE 1
WARREN MI 48090

PROJ MGR, M60 TANK DEVELOPMENT
USMC-LNO, MAJ. VARELLA 1
US ARMY TANK-AUTOMOTIVE CMD (TACOM)
WARREN MI 48090

PROG MGR, M113/M113A1 FAMILY
OF VEHICLES
ATTN DRCPM-M113 1
WARREN MI 48090

PROJ MGR, MOBILE ELECTRIC POWER
ATTN DRCPM-MEP-TM 1
7500 BACKLICK ROAD
SPRINGFIELD VA 22150

OFC OF PROJ MGR, IMPROVED TOW
VEHICLE
US ARMY TANK-AUTOMOTIVE R&D CMD
ATTN DRCPM-ITV-T 1
WARREN MI 48090

CDR
US ARMY EUROPE & SEVENTH ARMY
ATTN AEAGC-FMD 1
APO NY 09403

PROJ MGR, PATRIOT PROJ OFC
ATTN DRCPM-MD-T-G 1
US ARMY DARCOM
REDSTONE ARSENAL AL 35809

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MOFFIT FIELD CA 94035

CDR
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ATTN SDSTO-TP-S 1
TOBYHANNA PA 18466

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DRXMR-R 1
DRXMR-T 1
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CDR
US ARMY DEPOT SYSTEMS CMD
ATTN DRSDS 1
CHAMBERSBURG PA 17201

CDR
US ARMY WATERVLIET ARSENAL
ATTN SARWY-RDD 1
WATERVLIET NY 12189

CDR
US ARMY LEA
ATTN DALO-LEP 1
NEW CUMBERLAND ARMY DEPOT
NEW CUMBERLAND PA 17070

CDR
US ARMY GENERAL MATERIAL &
PETROLEUM ACTIVITY
ATTN STSGP-PW (MR PRICE) 1
SHARPE ARMY DEPOT
LATHROP CA 95330

CDR
US ARMY FOREIGN SCIENCE & TECH
CENTER
ATTN DRXST-MT1 1
FEDERAL BLDG
CHARLOTTESVILLE VA 22901

CDR
DARCOM MATERIEL READINESS
SUPPORT ACTIVITY (MRSA)
ATTN DRXMD-MD 1
LEXINGTON KY 40511

HQ, US ARMY T&E COMMAND
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