Contrails

EVALUATION OF A DETECTOR FOR FREE WATER IN FUEL

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FOREWORD

This report was prepared by Southwest Research Institute, San Antonio, Texas 78206, under Contract AF 33(615)-2327. The contract was initiated under Project Nos. 8169 and 3048, Task Nos. 816901 and 304801. The work was performed using Air Force facilities at Wright-Patterson Air Force Base and was administered by the Ground Support Branch and the Fuels, Lubrication, and Hazards Branch, Air Force Aero Propulsion Laboratory, Research and Technology Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The project engineer was Mr. Charles R. Martel (APFG).

This report covers one phase of the work performed under the subject contract between 1 March 1965 and 1 March 1966. This report was submitted by the authors on 18 March 1966 for publication as an AFAPL Technical Report. Contractor's identifying numbers for this report are Project No. 09-1663, Report No. RS-481.

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This technical report has been reviewed and is approved.

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ABSTRACT

A test apparatus for detecting small amounts of free water in jet fuels, which was developed by a Navy group, has been subjected to various evaluations. The test method consists of passing a fuel sample through a dye-coated test pad and then comparing the fluorescence level of the pad under ultraviolet illumination with that of permanent color standards. Based on static evaluations of the apparatus using standard water-in-fuel dispersions, the accuracy and precision are well within 5 mg/liter in the range of free water contents up to 20 mg/liter. In analyzing effluent fuel from filter-separator tests by this method, it often shows traces of free water when the standard Karl Fischer analyses indicate undersaturated fuel; this apparent discrepancy is caused by nonequilibrium conditions of water distribution in the effluent fuel. Based on rather limited data, the sensitivity of the free water detector is not affected adversely by normal concentrations of anti-icing additive, corrosion inhibitor, or iron oxide contaminant in the fuels. In comparison with the Karl Fischer method, the free water detector is far simpler and faster and is believed to give more meaningful results. The detector appears to be very suitable for use in preproduction testing of filter-separators and elements. It should also be adaptable for use in base fuel quality control by relatively unskilled personnel, particularly if equipment manufacturers can develop prepackaged pad holders and comparator viewing instruments suitable for field use. Under field conditions, direct line sampling through a water detector pad will be more reliable than the use of bottled samples. It is recommended that field trials and further laboratory evaluations should be performed to establish more firmly whether the variety of fuel contaminants encountered in the field will interfere with the sensitivity of the method.

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SECTION I

INTRODUCTION

The need for a rapid quantitative or semiquantitative method for the determination of free water* in jet fuels has existed for several years. Quality control of fuel handling operations in the field must be based on checking the amounts of free water and particulate matter carried along with the fuel, as well as on the installation of effective fuel cleaning facilities and their proper operation and maintenance. Methods for determining the amounts of free water and particulate matter are also needed to establish the performance of filter-separators and filter-separator elements in preproduction testing. The existing methods for determination of free water content of fuels have serious shortcomings, as will be discussed in Section II.

Any test method for determining the free water content of fuel, in order to be satisfactory for field use, should meet the following requirements: (1) The apparatus should be usable at the sampling site, with readout of results immediately or at most within a few minutes; (2) the apparatus should be simple and rugged in construction, so that it can be used successfully by relatively unskilled personnel; (3) the test method should be sufficiently sensitive to detect free water at concentrations of 5 mg/liter or lower, i.e., far below the limit of visual detection; (4) the apparatus should not require frequent calibration or maintenance; and (5) the cost of the apparatus and the operating cost per test should be low.

This report covers the evaluation of a free water detector apparatus and procedure developed by the Aeronautical Engine Laboratory of the Naval Aeronautical Engineering Center (AEL) and certain modifications worked out by personnel of the Air Force Research and Technology Division (RTD) in cooperation with an equipment manufacturer.

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*The term "free water" as used in this report refers to any undissolved water, whether suspended, "entrained," emulsified, or present in gross amounts as large drops or a bottom layer.

SECTION II

BACKGROUND

Modern fuel handling techniques for jet fuels place heavy reliance on the use of filter-separators, which are designed to coalesce and settle out any free water and also to reduce the content of particulate matter to an acceptably low level. Filter-separator malfunctioning may occur because of defective elements, faulty installation, inadequate maintenance, "poisoning" of the elements by traces of surfactant materials in the fuel, or a variety of other reasons; a full discussion of this subject is beyond the scope of this report. In any case, malfunctions do occur, so that test methods for detecting free water and particulate matter in fuel are essential. Malfunctions that permit the passage of gross quantities of free water or particulate matter can be detected by visual examination of fuel samples. However, malfunctions may permit the passage of relatively small amounts of contaminants, not detectable by visual examination, but nevertheless a serious hazard in aircraft operation.

The amount of solid particulate matter in fuels is determined by a relatively simple filtration and gravimetric analysis. Although this is not strictly a field method and requires the services of a capable laboratory technician, it has proven very useful in field operations and is being used routinely as a quality control method. Existing methods of analysis for free water have been less successful.

In the quality control of fuel handling operations, "dry" fuel is commonly considered to be fuel containing not more than 5 mg/liter of free water, as specified, for instance, in T.O. 42B-1-1C, "Quality Control of Fuels and Lubricants." However, this use limit is set largely by the precision limits of existing test methods; ideally, fuel leaving a filter-separator should have zero content of free water. In MIL-F-8901A, which governs the performance test requirements for filter-separators and elements, zero free water content is specified, defined in terms of the difference in total water content of the fuel leaving the test unit and that of the fuel after subsequent passage through an oversize "cleanup" filter-separator. In either case, the quantities of free water that are considered as excessive are small in relation to the dissolved water content of the fuel, which may average around 50 mg/liter in fuel handling systems*.

The only quantitative analytical method presently accepted for determining the water content of hydrocarbon fuels is a titration with Karl Fischer

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*During a recent survey[1] of JP-4 fuel handling systems under different climatic conditions, dissolved water contents ranging from 8 to 73 mg/liter were found.

reagent in accordance with ASTM D 1744-64. This method has several short-comings. First of all, there is difficulty in obtaining really precise results. The repeatability (95% confidence level) as listed in the ASTM standard is 11 ppm by weight, which corresponds to about 8 mg/liter (±4 mg/liter) in the case of JP-4. Even under the most carefully controlled conditions of sampling and analysis by well-trained personnel, using the most modern titration equipment and carrying out the analysis in an air-conditioned laboratory, the precision is very little better; under less favorable conditions, deviations of as much as 20 mg/liter may be encountered. Further, this method determines the total water content of the fuel, including both free and dissolved water. Therefore, the free water content must be obtained as the difference between the total water content of the actual fuel sample and that of a "saturated" sample of the same fuel that has been equilibrated over a water layer. Thus, the error of each determination can be cumulative when used to establish the free water content by difference. Many other factors affect the precision adversely. For example, it has been demonstrated[2] that the presence of the normal concentration of 0.1% anti-icing additive in JP-6 fuel can cause an error of about 10 mg/liter in determining the "saturation" value of the dissolved water content. Errors are also introduced by the phenomenon of "nonequilibration" of samples obtained in the field, i.e., the actual presence of free water accompanied by a dissolved water content that has not yet reached the maximum saturation value, so that the total water content may be lower than the saturation value even when free water is visibly present. Finally, the common practice of determining saturation values at a temperature other than that of the fuel sampling temperature can introduce serious errors[3]. Although some of these errors can be eliminated or accounted for by appropriate corrections and modifications of the "saturation value" procedure, this merely adds extra detail to a test procedure that is already considered quite difficult to perform satisfactorily. The Karl Fischer titration method was used for about two years as a check on fuel quality at Strategic Air Command bases, but the results were never considered sufficiently reliable to justify the immediate removal of suspect equipment from service, and SAC subsequently abandoned the use of the method for daily quality control.

Various simple devices have been proposed for the detection of free water in fuels, based on color change of a prepared chemical cartridge or filter, but most of these have been limited by various factors including lack of sensitivity in the lower ranges of free water concentration, on the order of 0-20 mg/liter. Devices with such a limitation are only a little better than visual observation, which can detect free water at only slightly higher concentrations.

Other approaches to the problem have included the use of instruments based on light scattering or other phenomena indicative of inhomogeneity of the fuel, and also the use of in-line cartridge elements that will shut off fuel flow automatically when excessive amounts of free water or particulate matter are encountered. An evaluation of some of these devices has been made

recently[4]. While the best of these devices are very promising and will undoubtedly find wide use in fuel handling operations, this will not eliminate the need for a sensitive and reliable method for measuring free water content in the field. The use of monitoring instruments or automatic shutoff devices probably will be limited to the "last chance" in the fuel handling sequence, i.e., immediately before the fuel enters the aircraft. There will still be a need for determining free water contents at other points farther back in the fuel system as a guide to operations, and also a need for an independent check on fuel quality at the aircraft skin as extra insurance against failure of the instrument or device being used at this point.

SECTION III

EQUIPMENT AND PROCEDURES

A. Free Water Detector

1. Principle of Operation

Test pads are prepared by coating paper filter pads with uranine (sodium salt of fluorescein), an orange dye that fluoresces strongly in aqueous solution under ultraviolet illumination. These test pads are prepared and maintained under anhydrous conditions until ready for test. When a fuel sample is to be tested for free water content, 500 ml of the fuel is filtered through the test pad, the excess fuel is removed by suction, and the test pad is compared under ultraviolet illumination with a series of color standards representing 0, 5, 10, and 20 mg/liter of free water content. When no free water is present, the pads when viewed under UV appear purple; free water in the fuel sample causes the pad to show a yellow fluorescence, with increasing intensity that depends on the amount of free water. Water contents that do not match any of the color standards are estimated by interpolation.

2. Viewing Box

The basic instrument used in all the evaluations reported herein was a prototype constructed by AEL, essentially equivalent to that specified in MIL-D-81227(WP), 9 July 1965, Detector, Free Water in Fuel, and the drawings referenced therein, AEL No. D-134 Sheets 1 and 2.

The instrument consists of an aluminum box, overall size approximately 10" × 5-1/2" × 4" high; the weight of the complete assembly is restricted to six pounds. The box contains a 6-watt "black" fluorescent tube with switch, starter, and ballast, as well as a reflector. The instrument is designed for plugging into a 120-volt, 60-cycle power source and is not of explosionproof construction. Four color-standard discs are mounted within the box, with provision for positioning a test pad and viewing it in comparison with the color standards.

3. Color Standards

In the early part of the work reported herein, the color standards provided by AEL consisted of test pads that had been exposed to known amounts of free water in fuel and then sealed in transparent moistureproof bags. These will be referred to as "temporary" standards. In the later stages of the work, permanent color standards were provided by AEL, similar to those specified in MIL-S-81282(WP), 9 July 1965, Standard, Free Water Detector. These are color-printed patches made with standard plates and standard inks,

corresponding in degree of fluorescence to 0, 5, 10, and 20 mg/liter free water.

4.   Test Pads

Each test pad consists of a 47-mm absorbent paper disc coated on one side with USP uranine in absolute ethanol solution; the optimum amount of uranine is 0.80-0.85 mg per pad. These pads are now covered by MIL-D-81248(WP), 30 July 1965, Detector Pad, Free Water. The "nutrient pads" supplied by manufacturers of membrane filters for use in microbiological work are suitable for this use. The test pad specification requires that the pads be dry before coating and that the coating operation be performed at 70-80°F and a relative humidity of 30% or lower. The pads thus prepared are sealed individually in moistureproof bags conforming to MIL-B-117, Class E, Type II. Samples of the packaged pads are selected for exposure tests under high-humidity conditions. The test pad specification also requires that the uranine content must be 0.80-0.85 mg per pad, based on fluorometric analysis of the material extracted from a test pad.

During the later stages of the evaluation reported herein, some test pads were furnished by two manufacturers; it is understood that these were prepared by the general methods given in the current specification. One manufacturer furnished 37-mm pads for use in field monitor equipment, and the other furnished standard 47-mm pads. In both cases, the pads were individually packaged by the supplier in heat-sealed moistureproof bags that presumably conformed to the MIL-B-117 specification.

Most of the pads used in the evaluations reported herein were prepared by SwRI personnel, and it will be understood that such pads were used in obtaining all data reported except where indicated otherwise. The preparative method, which followed that recommended by AEL at the time, is given in abbreviated form in the following paragraph.

Absorbent pads, "Millipore AP-10" or equivalent, are oven dried for 15 minutes at 220°F and kept in a desiccator until ready for spraying. A solution consisting of 0.5% uranine in absolute ethanol is prepared and placed in a hand sprayer*. The solution is applied by spraying a pad until it is uniformly coated with the desired amount of uranine; multiple sprays of short duration have been found to be more effective in achieving uniform coverage. The sprayed pads are stored in a desiccator, protected from light,

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*In the early stages of the program, a chromatographic sprayer was used. Later, all pads were prepared using a "Jet Pack Unit" No. 50-SS (aerosol-pressurized) with four sprays of about 0.5 sec each from a distance of 14 inches.

until ready for use. Randomly selected test pads are checked for uranine content by extracting the dye with water and comparing the extract with standard solutions of uranine, using a "Spectronic 20" colorimeter at 510 mμ wavelength. The quality of the pads can also be checked by filtering fuel containing known quantities of free water, then comparing the pads against the color standards. Both of these quality checks are destructive test methods.

This procedure conforms generally to the test pad specification except that this procedure does not place any limit on spray-room relative humidity. In our evaluations, it was found necessary to control ambient humidity to 50% or lower in order to obtain satisfactory results; the test pad specification now requires 30% or lower.

5.   Test Pad Holders

Most of the work involved the use of a "Millipore No. XX20 047 20" stainless steel filter holder for 47-mm pads, mounted on a filter flask. After drawing through a 500-ml sample of fuel, the test pad was removed as soon as the filter was sucked dry and immediately compared with the color standards under ultraviolet illumination. Using this filter holder, the "effective diameter" of the test pad is approximately 35 mm, giving an effective filtration area of about 960 mm^2. The smaller 37-mm test pads can also be used in this same equipment, provided care is taken in centering the pads. It is understood that the AEL development work on the free water detector was performed primarily with line-type holders for 47-mm pads and that the effective diameter of the pads in these holders is also approximately 35 mm.

When using 37-mm pads in "field monitor" type filter holders, the effective diameter is likewise about 35 mm. However, the use of a certain type of flow diffuser inside these monitors did lead to some reduction in effective filtration area, as will be discussed later in this report. In any case, whenever a filter mounted in a field monitor was used for test, a 500-ml fuel sample would be passed through the monitor, normally installed inside a bomb sampling kit attached directly to the fuel line being sampled; then the monitor would be taken to the laboratory for removal of excess fuel by suction, disassembly, and rating of the test pad.

B.   Test Samples and Procedures

All of the evaluations reported herein were performed using samples of JP-4 fuel. This fuel contained no additives unless otherwise stated.

For static evaluations of treated test pads, standard dispersions of free water in fuel were prepared by saturating a fuel sample over water for a minimum of 16 hours at room temperature, then transferring 500 ml of the saturated fuel to a Waring blendor and agitating for two seconds after adding the required amount of free water. This dispersion was passed immediately through a test pad, using the stainless steel 47-mm holder and filter flask.

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Line samples were available from a number of filter-separator tests being run in a single-element evaluation program. The effluent fuel was sampled and tested regularly, using the Karl Fischer titration for comparison with the results obtained with the free water detector. A similar schedule of effluent sampling and testing was maintained in connection with a study of dyed fuel performance in the filter-separator system of an F-6 refueling semi-trailer. In these two evaluation programs, the detector tests were run primarily with bottled samples* and laboratory filtration. However, in the later stages of the single-element test program, 37-mm pads in field monitors were used in a bomb sampler attached directly to the fuel effluent line.

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*In drawing bottled samples, the usual precautions were taken with regard to preflushing the sampling line thoroughly and prerinsing the sample bottle at least twice with test fuel.

SECTION IV

PREPARATION AND STATIC EVALUATION OF TEST PADS

The initial work on treated pads received from AEL was performed when the equipment and the pad preparation were still in the development stage; the results were quite scattered and will not be reported here. All of the work reported herein is based on uranine-coated pads prepared in the manner outlined in Section III.

The first evaluation of pads prepared by AEL using the current technique was performed with standard dispersions of free water in saturated JP-4 (Waring blendor), using the "temporary" color standards. The JP-4 was saturated by storing over water for 24 hours at ambient temperature that varied from 75 to 82° F; the fuel temperature was 82° F at the time the 500-ml samples were removed for addition of free water in the Waring blendor. The following results were obtained in comparison with Karl Fischer titrations:

| | Free water, mg/liter | | | Total water |
| | Free water detector | | Karl | content, mg/liter |
Added	Between	Estimated	Fischer*	(Karl Fischer)*
0	0/0	0	0	106
5	0/5	3	4	110
10	5/10	8	8	114
20	10/20	17	24	130

These preliminary results showed reasonably close agreement with the Karl Fischer data. It was noted that the blending time in preparing the dispersions was quite critical, and it is thought that sample temperature rise during blending may have been responsible for the low detector result on the 20 mg/liter dispersion. For further studies, the water blending procedure was standardized with a two-second blending time as indicated in Section III.

The initial work by SwRI on preparation of test pads was performed using a chromatographic sprayer, trying different combinations of number and duration of sprays. Although the uranine contents of these pads were not determined quantitatively, they appeared to be excessive, based on visual comparison with AEL-prepared pads. Also, these freshly prepared pads gave low readings (0-7 mg/liter) on standard 10 mg/liter water-in-fuel

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*These Karl Fischer analyses were performed with very careful control of ambient conditions and all details of sample handling and titration. Each of the results reported represents the average of at least three determinations.

dispersions. Pads from another batch, prepared with shorter or fewer spray periods, gave correct readings on 20 mg/liter dispersions. There was an overall trend toward better accuracy for the pads with fewer and/or shorter spray periods, i.e., with lower uranine contents.

The next pads to be evaluated were prepared with two sprays of one or two seconds each. Some of these pads were checked for uranine content by colorimetric analysis of the extracted dye, and the others were tested with standard water-in-fuel dispersions. The uranine contents of these pads, in comparison with pads received from AEL at that time, were as follows:

2 sprays, 2 sec each:	1.15, 1.08, 1.36 mg	Average 1.20 mg
2 sprays, 1 sec each:	0.84, 0.60, 0.74, 0.61, 0.74, 0.71, 0.66 mg	Average 0.68 mg
AEL-prepared pads:	0.57, 0.74 mg	Average 0.66 mg

Thus, neither the SwRI-prepared pads nor the AEL-prepared pads fell within what is now the specification range of 0.80-0.85 mg uranine per pad.

The remaining pads from the lot prepared by SwRI were evaluated with standard water-in-fuel dispersions, with the following results:

	Free water, mg/liter	
Pad preparation	Added	Indicated
Two 2-sec sprays	20	20, 20, 23*
Two 1-sec sprays	20	20, 20
Two 2-sec sprays	10	10, 10
Two 1-sec sprays	10	10, 8*, 10
Two 1-sec sprays	5	5, 5

From these results, it appeared that both groups of SwRI-prepared pads were equally satisfactory in spite of the very considerable difference in uranine contents of the two groups, where the extreme range was from 0.50 to 1.36 mg per pad based on analysis of random samples from each group.

A brief investigation was made of the possible effect of a yellow dye on the accuracy of the free water detector. This dye, "Automate Yellow No. 8," has been proposed for addition to JP-4 fuel as a leak detector in connection

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*Estimated values.

with certain aircraft operational problems; the proposed concentration is
1 lb/1000 gallons (120 mg/liter). Standard free water dispersions were pre-
pared from dyed and undyed JP-4 fuel and evaluated using SwRI-prepared test
pads, with the following results:

Free water added, mg/liter	Free water indicated, mg/liter	
	Dyed fuel	Undyed fuel
0	---	0
5	5	5
10	9-10	9-10

These data indicate that the use of the dye in JP-4 fuel should have no effect
on the accuracy of the free water detector. Subsequent studies on dyed fuel
are reported in Section V.

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SECTION V

LINE SAMPLE EVALUATIONS

A.   General

In view of the encouraging results from the preliminary evaluations, the free water detector was incorporated as a supplementary analysis in certain development and evaluation programs being conducted by SwRI at the Wright-Patterson AFB fuel filtration facility. These included a single-element filter-separator program and an evaluation of the effect of a fuel dye on filter-separator operation using an F-6 refueling semitrailer. The use of Karl Fischer titrations was continued, with the free water detector being used as an additional check. Since the primary objective of these programs was not the evaluation of the free water detector, the data had to be taken as they came; i.e., it was not possible to adjust the test program to provide a broad-range evaluation of the detector under a variety of conditions. Nevertheless, the data obtained did provide further checks on the reliability and accuracy of the free water detector and pointed out some possible problems to be resolved for successful application of the detector in field fuel quality control.

Several lots of test pads were prepared for these programs, using the AEL-recommended method of preparation (Section III-B). The pads were prepared in lots sufficient to cover estimated requirements for one month of testing and kept in desiccated storage up to the time of use. The treated pads were sampled periodically for checking the accuracy of readings given with standard water-in-fuel dispersions.

In most of this work, the line samples of fuel were taken in bottles and passed through a 47-mm test pad in the laboratory holder; in later work, direct line sampling through a 37-mm detector pad was adopted for part of the testing.

B.   Precision of Karl Fischer Test Results

In order to assess properly the comparative values obtained with the detector vs those obtained by the Karl Fischer method, it is necessary to arrive at some reasonable estimate of the precision of the Karl Fischer method as employed in our testing.

So far as the majority of the work was concerned, the absence of anti-icing additive in the test fuel eliminated this one source of error in the "saturation values" that were obtained. Therefore, the major deviations were derived from the scatter of results in the Karl Fischer test itself and from the fluctuations in line sampling temperature.

The ASTM statement of repeatability of the Karl Fischer results cites a value of 11 ppm by weight, which is approximately 8 mg/liter (± 4 mg/liter)

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in the case of JP-4 fuel. However, this value is based on total water contents from 50 to 1000 ppm, and it may be argued that the absolute deviations will be less in the lower end of this range, in which all of our determinations were made. Lacking any statistically reliable data to confirm or deny this point, only an opinion can be offered. We feel that the absolute deviations will be much the same regardless of the level of total water content, since the absolute errors introduced by gain or loss of water during sample handling and those introduced by pickup of atmospheric moisture in the titration apparatus should be largely independent of total water content. At any rate, in the discussion of data on line samples, it is assumed that in our work the precision of the Karl Fischer analysis in terms of repeatability is 6 mg/liter, i.e., slightly better than that cited by the ASTM.

The water content of water-equilibrated fuel, determined by Karl Fischer titration and used as the "saturation value," is subject to a further error in that the line sampling temperature is normally controlled to ±2°F, whereas all saturation values read from the solubility-temperature curve are based on the average fuel line temperature for the entire test period. For the fuels used in this program, the change of water solubility with temperature was about 0.5 mg/liter per °F; therefore, the 4°F error in sampling temperature introduced an error of about 2 mg/liter that must be applied to the saturation value.

Based on a repeatability of 6 mg/liter for the test sample and 8 mg/liter for the saturation sample, the repeatability of the difference value (representing either free water content or "undersaturation" of the fuel sample) is calculated to be 10 mg/liter, assuming normal distribution of errors in all cases. This estimated repeatability of 10 mg/liter (±5 mg/liter) is used in all subsequent discussion of results.

It must be emphasized again that, apart from any question of repeatability, the interpretation of Karl Fischer results on line fuel samples is complicated by the fact that the samples taken in filter-separator tests are often in a nonequilibrated state, owing to the rapid passage of fuel from the point of water injection into the filter-separator and then into the effluent sampling line. In the particular test loop used in this program, fuel travel is very rapid between the point at which excess water is injected and the filter-separator test unit; the residence time in this line is on the order of two or three seconds. Fuel residence time in the filter-separator housing (after passing through the element and separator screen) and in effluent lines up to the sampling point is estimated to be not more than 20 seconds. In the few seconds of flow time that elapse between the water injection and passage of the fuel through the filter-separator element, it is not possible for the fuel to become water-saturated (despite the injection and fine dispersion of excess water), owing to the relatively slow transfer of water molecules through the liquid-liquid interfaces. After passage through the filter-separator element and separator screen, the amount of free water present is ideally zero, but

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small amounts of free water (in the range of a few mg/liter) may be present
in the fuel flowing out of the filter-separator housing. This free water may
often be present in the form of scattered individual droplets rather than in the
form of the very fine dispersion fed into the test unit; hence, the liquid-liquid
interface area presented for molecular transfer is very small. Under these
conditions, the 20 seconds elapsing between the working section of the filter-
separator and the sampling point is likewise insufficient to establish equilibrium
saturation conditions.

The incoming fuel normally is well below saturation, with the actual
value depending on the ambient temperature and humidity and the residence
time of the fuel in the storage tank. Assuming an incoming fuel with an actual
dissolved water content of 40 mg/liter and a saturation value of 80 mg/liter,
the fuel would have to pick up an additional 40 mg/liter to reach saturation.
The short periods of time that are involved do not permit this equilibration to
go to completion, so that the effluent fuel typically is still undersaturated.
Now, if the filter-separator is not functioning at 100% efficiency, small amounts
of free water will still be present in the effluent fuel. For example, if the fuel
saturation process goes only halfway to completion, the effluent fuel will con-
tain 60 mg/liter of dissolved water and (say) 3 mg/liter of free, undissolved
water. The Karl Fischer titration of this sample should indicate a total water
content of 63 mg/liter, i.e., 17 mg/liter below the saturation value. This is
normally interpreted as indicating the absence of free water. Thus, it can
be seen that the Karl Fischer titrations, even if they were absolutely precise,
could not detect the presence of free water under these conditions, and should
not be expected to check in all cases the results of an independent determina-
tion of free water content in line samples, as represented by the free water
detector.

This question of nonequilibration of line samples has been discussed in
some detail since it has an important bearing on the interpretation of the test
data to be presented. The phenomenon has been observed repeatedly by per-
sons engaged in filter-separator testing, although it does not appear to have
been well documented. There have also been some indications[1] that similar
situations can occur in field operation of filter-separators in refueling opera-
tions.

C.   Samples from Single-Element Filter-Separator Tests

During the period covered by this report, a single-element test loop
was being operated in a program aimed at the development of realistic methods
for life-testing elements. The test section consisted of a housing with a single
20-gpm filter-separator element conforming to the military standard design
requirements (MIL-F-52308) and a single military-design canister of Teflon-
coated screen as the permanent separator stage. Elements from a single
manufacturer were used in all of the tests reported herein. It should be
mentioned that performance capability of the military-design elements at

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20 gpm is still somewhat of a controversial question, and that in our program these elements probably were being tested at very nearly the upper limit of their capability.

JP-4 fuel without additives was used throughout these tests. Each test was run to 125 hours maximum. The procedure in most of these tests consisted of injecting red iron oxide upstream of the test section at the rate of 0.33 mg/liter of fuel throughout the entire test period. The red iron oxide, which was the Fisher I-116 grade normally used in filter-separator testing, was injected in the form of a dilute slurry with JP-4 fuel only; this slurry was recirculated continuously against back pressure and also stirred, in order to obtain a very fine dispersion of the originally aggregated particles. Water was injected upstream of the test section at 1% of fuel flow rate for 10-minute periods at the beginning and end of the first three eight-hour operating days, and thereafter at the end of each operating day. Samples were taken midway of the water injection periods and analyzed for particulate matter and water content, using the Karl Fischer titration as the primary check on water content. After the free water detector had been checked out in the static tests described in Section IV, its use was introduced into the filter-separator test program to obtain additional information.

Two of the more recent tests in this filter-separator test program were run according to the life-test procedure of MIL-F-8901A, which differs from the procedure just described in that the red iron oxide is injected at a much faster rate and without good dispersion; the element is loaded with red iron oxide to 10-psi pressure drop at the start of each eight-hour operating period. Water is injected at 0.5% of fuel flow rate for the first hour of each operating period*. Also, the sampling schedule differs markedly, in that 8901A life testing requires sampling and analysis of the effluent fuel every ten minutes during each water injection period, and also every four hours of test.

In most of the tests reported herein, a "Totamitor" was used in the effluent fuel line as an additional check on the passage of contaminants. This instrument consists of a sensing unit installed in the main fuel flow line, with a light source and photocell arranged so as to pick up light scattered by any insoluble particles dispersed in the fuel; the photocell output is amplified and transmitted to suitable readout instruments and/or alarm. The Totamitor was set so that the readout scale would nominally give ppm free water (approximately mg/liter) as a direct reading. However, the instrument responds to both solid and liquid contaminants, and the readings obtained at a given contamination level are influenced by the degree of dispersity of the contaminant[4].

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*During the first eight-hour operating period, the water injection period is usually longer than one hour, since it is continued along with the red iron oxide injection until the pressure drop reaches 10 psi; this may require two to three hours when the test is first started.

Therefore, the Totamitor readings must be regarded as only a semiquantitative measure of contaminant level.

The first use of the free water detector was made in Test No. 06 (continuous injection of red iron oxide). In this test, a water injection after 38.4 hours of operation yielded an effluent sample with a Karl Fischer water content of 136 mg/liter, in comparison with a saturation value of 77 mg/liter. As no free water could be detected visually, a gross error in the Karl Fischer titration result was suspected, particularly since it had been performed under high-humidity conditions. This situation was corrected before proceeding. The next day, a repeat water injection was performed immediately after startup, and the fuel effluent was sampled and tested with a detector pad, which indicated 1 to 2 mg/liter free water. An hour later, water injection was again performed, taking samples simultaneously for Karl Fischer and free water detector tests. The Karl Fischer indicated 58 mg/liter total water, i.e., 19 mg/liter below saturation, whereas the detector showed a trace of free water, estimated at less than 1 mg/liter. These check analyses were performed periodically throughout the remainder of the test, with the Karl Fischer continuing to indicate water contents 15-24 mg/liter below saturation, but with the detector occasionally indicating traces of free water, less than 1 mg/liter.

At the termination of this 125-hour test, water was injected at increasing rates until definite failure occurred by passage of free water into the effluent fuel. Totamitor readings were also available at this time. The following comparative data were obtained as the water injection rate was increased:

Water content by Karl Fischer, mg/liter			Free water content by detector, mg/liter	Totamitor reading
Total	Satn	Free		
57	77	-20	Trace (<1)	10
93	77	16	12-14	20

In the light of later findings, it appears probable that the first sample, taken when the Totamitor reading reached 10, may have represented mainly the passage of solids disoldged from the element by the increased water flow, whereas the second sample represented the passage of definite amounts of free water and also some solids. In any event, the Karl Fischer and detector results were in good agreement for this second sample.

At the end of the next test (No. 07), which was terminated at 64.9 hours because of excessive pressure drop, failure was again induced artificially by increasing the water injection rate until a Totamitor reading of 10 was obtained. At this time, the Karl Fischer results indicated 13 mg/liter below saturation, while the detector indicated a free water content of 2-3 mg/liter.

In Test No. 07 and all subsequent tests, both the free water detector and Karl Fischer analysis were applied regularly to samples taken during water injection periods, as listed in Table 1. Although it is not the purpose of this report to discuss the overall test program, some remarks on the general outcome will be helpful in interpreting the data on water content. Test Nos. 07 and 08 were terminated prematurely because of excessive pressure drop, with no evidence of the passage of solids or water. Test Nos. 09, 11, 13, 14, and 15 were terminated prematurely because of transient passage of red iron oxide into the fuel effluent at the start of the water injection periods*. Test No. 12 showed the same behavior, but was continued for the full 125 hours. Test No. 10 was terminated early for inspection of the underground fuel tanks, the results of this test having indicated satisfactory performance up to the time of termination. Test Nos. 16 and 17 were run with a different method and schedule of solids and water injection, corresponding to the life test specified in MIL-F-8901A; these tests were also terminated prematurely because of the same type of transient passage of red iron oxide encountered previously. For these two tests, samples were taken every 10 minutes during water injection periods for determination of water (AEL detector) and solids contents; however, the only data shown in Table 1 are those for which comparative Karl Fischer data are available.

The data of Table 1 show that all total water contents (Karl Fischer) were below the corresponding saturation values, although there were several instances in which the indicated undersaturation was within or near the estimated precision limit of 10 mg/liter. The free water detector gave mainly zero readings and indicated at most a trace of free water in some samples (excluding the induced failures at the ends of Test Nos. 06 and 07). Of the nine instances in which the detector indicated a trace of free water, three of the corresponding Karl Fischer undersaturation values were definitely less than the estimated repeatability, one value was close to this limit, and five were definitely greater. These five cases must be explained on the basis of nonequilibrium conditions in the flow system, as discussed previously.

It is of importance to note that the detector and Karl Fischer results were in agreement that no really dangerous quantities of free water ever passed into the effluent fuel under the regular operating conditions. Likewise, the Totamitor gave no indications of contaminant passage during the regular sampling. The only comparative data under more severe conditions were those from the induced failures that were discussed previously.

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*The transient passage of iron oxide into the fuel effluent upon starting a water injection was detected by the Totamitor readings and confirmed by analysis of effluent fuel taken at such times. In general, the passage of iron oxide into the effluent had ceased by the time of the regular sampling, several minutes after the start of water injection. Therefore, the data shown in Table 1 do not reflect these transient conditions.

TABLE 1. ANALYSES OF EFFLUENT FUEL IN SINGLE-ELEMENT TESTS

Test time, hr	Element ΔP, in. Hg	Test results on effluent fuel[a]		
		Undersaturation, mg/liter (Karl Fischer)	Free water, mg/liter (detector)	Particulate matter, mg/liter
Test No. 07, water saturation value 73 mg/liter[b]				
0.0	3.9	15	0	0.00
8.0	4.5	11	0	0.03
15.6	6.5	13	0	0.00
23.6	8.2	22	0	0.01
31.6	10.9	20	0	0.00
39.6	11.8	26	0	0.00
47.6	21.8	24	0	0.00
55.6	47.6	21	0	0.05
63.6	67.4	32	0	0.00
64.9	>100	15	0	0.00
Induced failure		13	2-3	---
Test No. 08, water saturation value 77 mg/liter[b]				
0.0	5.0	46	0	0.02
8.0	7.1	33	0	0.01
16.0	9.0	32	0	0.00
24.0	11.5	32	0	0.00
32.0	16.3	20	0	0.02
38.0	17.0	37	0	0.02
46.0	20.6	41	0	0.01
54.0	29.6	39	0	0.00
62.0	34.9	26	0	0.00
70.0	52.0	33	0	0.23
76.9	80.2	22	0	0.01
Test No. 09, water saturation value 65 mg/liter[b]				
0.0	3.2	11	0	0.00
8.0	5.6	11	0	0.01
16.1	7.2	10	0	0.06
24.0	7.7	1	<1[c]	0.00

See final page of table for footnotes.

TABLE 1. ANALYSES OF EFFLUENT FUEL IN
SINGLE-ELEMENT TESTS (Continued)

Test time, hr	Element ΔP, in. Hg	Test results on effluent fuel[a]		
		Undersaturation, mg/liter (Karl Fischer)	Free water, mg/liter (detector)	Particulate matter, mg/liter
Test No. 10, water saturation value 65 mg/liter[b]				
0.0	4.3	9	<1[c]	0.00
7.7	5.0	5	0	0.07
15.7	7.3	---	0	---
16.8	7.2	7	0	0.01
Test No. 11, water saturation value 65 mg/liter[b]				
0.0	4.6	10	0	0.00
6.8	5.3	6	0	0.02
11.5	5.8	7	0	0.10
Test No. 12, water saturation value 65 mg/liter[b]				
0.0	4.6	11	<1[c]	0.00
6.8	5.0	3	<1[c]	0.00
14.8	5.3	20	0	0.00
23.8	6.4	9	0	0.34
31.9	7.6	13	0	0.02
39.9	9.3	22	0	0.00
48.5	9.8	14	0	0.00
56.5	12.5	9	0	0.00
64.6	16.1	10	0	0.17
72.8	20.6	17	0	0.13
80.8	23.3	16	0	0.00
88.8	25.4	16	<1[c]	0.48
91.4	21.3	12	0	0.16
99.4	30.8	10	0	0.06
107.4	36.0	14	0	0.00
111.5	38.4	25	0	0.38
118.5	47.0	14	0	0.07
125.5	55.6	12	0	0.37

See final page of table for footnotes.

TABLE 1. ANALYSES OF EFFLUENT FUEL IN
SINGLE-ELEMENT TESTS (Continued)

Test time, hr	Element ΔP, in. Hg	Test results on effluent fuel[a]		
		Undersaturation, mg/liter (Karl Fischer)	Free water, mg/liter (detector)	Particulate matter, mg/liter
Test No. 13, water saturation value 73 mg/liter[b]				
0.0	4.5	21	0	0.39
8.0	4.4	16	0	0.08
16.0	5.1	13	0	0.03
24.0	5.9	29	0	0.26
32.0	6.5	27	0	0.03
40.0	7.4	19	0	0.03
48.0	8.4	47	0	0.04
56.0	11.2	27	0	0.29
64.0	14.5	39	0	0.37
73.0	15.1	18	0	0.48
Test No. 14, water saturation value 73 mg/liter[b]				
0.0	4.1	29	0	0.14
8.0	4.9	23	0	0.05
16.0	5.9	20	0	0.27
22.9	7.0	25	<1[c]	0.52
29.5	8.6	22	<1[c]	0.00
Test No. 15, water saturation value 73 mg/liter[b]				
0.0	3.5	19	0	0.30
6.0	4.5	23	0	0.09
14.5	5.5	20	0	0.33
24.0	7.1	20	0	0.42
32.0	8.2	11	0	0.22

See final page of table for footnotes.

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TABLE 1. ANALYSES OF EFFLUENT FUEL IN
SINGLE-ELEMENT TESTS (Continued)

Test time, hr	Element ΔP, in. Hg	Test results on effluent fuel[a]		
		Undersaturation, mg/liter (Karl Fischer)	Free water, mg/liter (detector)	Particulate matter, mg/liter

Test No. 16, water saturation value 73 mg/liter[b]

Test time, hr:min				
0:00	4.2	40	0	0.11
0:10	5.7	25	0	0.32
1:00	8.4	15	0	0.61
2:27	20.0	11	0	0.94
8:00	8.2	34	0	0.23
8:30	18.4	15	0	0.48
9:00	18.2	16	0	0.25
16:00	12.3	48	0	0.18
16:30	20.7	23	0	0.09
17:00	20.7	19	0	0.15
24:00	16.6	49	0	0.22
24:30	23.1	32	<1[c]	0.60
25:00	23.6	32	0	0.24
32:00	19.0	48	0	0.06
32:30	24.6	28	0	0.13
33:00	24.8	35	0	0.30

See final page of table for footnotes.

TABLE 1. ANALYSES OF EFFLUENT FUEL IN SINGLE-ELEMENT TESTS (Concluded)

Test time, hr:min	Element ΔP, in. Hg	Test results on effluent fuel[a]		
		Undersaturation, mg/liter (Karl Fischer)	Free water, mg/liter (detector)	Particulate matter, mg/liter
Test No. 17, water saturation value 73 mg/liter[b]				
0:00	4.5	47	0	0.18
0:30	6.8	20	0	0.26
1:00	8.7	27	0	0.25
2:33	20.0	23	0	0.30
8:00	7.1	45	0	0.12
8:30	18.5	24	0	0.26
9:00	17.7	25	0	0.24
16:00	10.9	38	0	0.20
16:30	21.2	21	0	0.34
17:00	21.3	21	0	0.23
24:00	15.2	44	0	0.04
24:30	23.5	26	<1[c]	0.20
25:00	23.4	25	0	0.01
32:00	18.2	39	0	0.00
32:30	25.2	18	0	0.00
33:00	25.5	21	0	0.00

(a) Effluent fuel was sampled midway in each water injection period in Test Nos. 7-15. Sampling schedule in Test Nos. 16 and 17 conformed to MIL-F-8901A.

(b) Water saturation values correspond to effluent fuel temperature, which was $69 \pm 1°F$ for Test No. 07 and $75 \pm 2°F$ for other tests.

(c) Only a few fluorescent specks visible.

It is also of interest to note that the functioning of the free water detector apparently was not hampered by the presence of particulate matter in amounts up to 0.9 mg/liter. Based on the appearance of the filter pads used in determining particulate matter, it consisted predominantly of the red iron oxide that was injected, although in some cases extraneous matter introduced from elsewhere in the test loop plumbing may have been included.

Thus, the data accumulated during these tests indicate that the detector did not give any spurious indications of significant amounts of free water, based on a large number of tests, and that the detector gave good agreement with the Karl Fischer results in the single instance of free water content in the 12-16 mg/liter range.

A significant fact not evident from Table 1 is that positive indications of free water by the detector occurred much more frequently when it was used in direct line sampling (field monitor) than when it was used on bottled samples. The data shown in Table 1 include only those samples for which comparative Karl Fischer and detector data were available. The sampling schedule was such that these particular detector tests were all run on bottled samples. However, additional water detector tests were run at intermediate times, and all of these were run directly from the line in field monitors. Considering only the samples taken during water injection periods, the detector results came out as follows:

	On-line	Bottled
Number of tests showing free water content of:		
Zero	18	20
0-1	18	2
1-2	3	None

From these data, the trend toward higher detector readings in on-line use is quite evident. This is understandable in the light of the previous discussion of the nonequilibrated state of fuel effluent samples. With small amounts of free water in the effluent, less than 5 mg/liter, the sample represents a "moving target," for which the actual free water content will decrease progressively by dissolving in the fuel phase. Even though every effort was made toward fast analysis of the bottled samples, the time interval between drawing the bottled sample and passing it through the laboratory-mounted test pad amounted to some 60-120 seconds, which is quite long in relation to the residence time of the fuel in the flow system between the test element and the sampling point (estimated to be not more than 20 seconds).

Further confirmation of this trend was obtained in a recent single-element test (likewise under MIL-F-8901A conditions), in which samples were drawn simultaneously from sampling valves located opposite each other in the

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effluent line. One was drawn directly through a test pad mounted in a field monitor, while the other was drawn into a bottle, rushed into the laboratory, and passed through a laboratory-mounted test pad. This comparison was repeated twice, and in each case the field-monitor pad showed a trace of free water (less than 1 mg/liter) and the laboratory-mounted pad showed zero free water.

For additional verification of the apparent loss of free water caused by redissolving in bottled samples, the fuel effluent stream was sampled during an induced failure at the end of another recent single-element filter-separator test. By increasing the water injection rate considerably, small amounts of free water started coming through in the fuel effluent, as evidenced by Totamitor readings of 2 to 7 (nominally corresponding to 2 to 7 ppm of free water). Three sets of samples were taken during this induced failure, each set consisting of three samples drawn simultaneously from closely adjacent points in the high-velocity section of the fuel effluent stream. One sample was drawn directly through a 37-mm detector pad mounted in a field monitor with flow diffuser, one sample was drawn into a glass bottle after the usual prerinses, and the third sample was drawn into a polyethylene bottle; sample volume was 500 ml in all cases. The bottled samples were rushed into the laboratory and passed through 47-mm detector pads. The following results were obtained on the three sets of samples:

Set No.	I	II	III
Totamitor reading	5	5	7
Free water, mg/liter			
Direct line sampling	2-3	2-3	5-7
Glass bottle	0-1	0-1	0-1
Polyethylene bottle	0-1	0-1	0-1

It can be seen that the direct line sampling gave results that were in general accord with the Totamitor readings, while the bottled samples gave results that were lower, never exceeding the 0-1 mg/liter range. The glass and polyethylene bottles gave identical results, indicating that the "loss" of free water in bottled samples is not due merely to wall adsorption effects, as has been suggested in the past. A fuel sample corresponding to Set I was also checked for total water content (Karl Fischer), which was indicated to be 53 mg/liter vs a saturation value of 71 mg/liter, i.e., an undersaturation of 18 mg/liter. Thus, these results provide confirmation that line sampling is a "must" for obtaining accurate detector results and that the amount of free water that can be lost in bottled samples is quite sizable when the fuel phase is undersaturated. It is worth emphasizing that, in the work just cited, the laboratory determinations on the bottled samples were performed within about a minute after drawing the samples, which is a condition that could hardly be maintained in field quality control. In field operations, the considerably longer

hold times between sampling and analysis would favor the possibility of redissolving major amounts of free water originally present, given either a substantially undersaturated fuel or a sample warmup in the time interval between sampling and analysis.

D.   Samples from Dyed Fuel Evaluation in F-6 Refueler

Additional comparisons between the free water detector and the Karl Fischer method were obtained during a brief evaluation of the effect of a fuel dye ("Automate Yellow No. 8") on the water-removing capability of one of the filter-separators of an F-6 refueling semitrailer. These tests were run using the same type of additive-free JP-4 fuel used in the single-element program, and also on JP-4 obtained from regular Wright-Patterson AFB stock, containing 0.08% anti-icing additive and presumably a corrosion inhibitor, since Air Force procurements during this period did require such material*. Both of these fuels were tested without and with dye (1 lb/1000 gal).

Each test consisted of recirculating fuel for 30 minutes from the refueler tank through one of the 300-gpm filter-separators and back to the tank. Fuel flow rates were 60-90 gpm, being limited by the engine and pump capability and control settings of the system. Water was injected into the pump suction at 1.5 gpm during the fuel circulating period. Effluent fuel samples were taken in bottles every five minutes and passed through the laboratory-mounted filter unit within one minute after drawing. Effluent fuel samples were also taken every ten minutes for Karl Fischer analysis. No solid material was injected at any time, and all effluent fuel samples showed particulate matter contents of not more than 0.05 mg/liter.

The comparative data on water contents of fuel effluent samples are listed in Table 2. The Karl Fischer data all showed undersaturation. However, the detector data indicated that fairly significant amounts of free water were present in some of the effluent samples; these occurrences were quite random and could not be correlated with type of fuel or the presence or absence of dye. All of the detector tests indicating free water in the range of 2 to 4 mg/liter occurred at sampling periods when no Karl Fischer data were obtained, which is extremely unfortunate in terms of the desired comparison. There may be some significance in the fact that, among the points of direct comparison (0, 10, 20, and 30 minutes), the three positive indications given by the free water detector corresponded to relatively small undersaturation values, approaching the estimated repeatability of the Karl Fischer results. This is illustrated by the following comparison:

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*The particular corrosion inhibitor in this fuel was not identified. Based on previous data on base fuel handling[1], it is probable that intermixing of fuels from different suppliers would result in the presence of more than one corrosion inhibitor in the fuel.

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TABLE 2. WATER CONTENT DATA FROM F-6 REFUELER TESTS

	Water (mg/liter) at times indicated (min)						
	0	5	10	15	20	25	30
1. Nonadditive JP-4 without dye							
Undersaturation (K-F)	24	---	24	---	25	---	11
Free water (detector)	0	0	0	0	0	0	0
2. Nonadditive JP-4 with dye[a]							
Undersaturation (K-F)	16	---	16	---	18	---	29
Free water (detector)	0	0	0-1	0	0	1-2	0
3. Nonadditive JP-4 with dye[b]							
Undersaturation (K-F)	11	---	24	---	24	---	21
Free water (detector)	1-2	2-3	0	3-4	0	0	0
4. Regular JP-4 without dye							
Undersaturation (K-F)	28	---	29	---	22	---	25
Free water (detector)	0	0	0	2-3	0	0	0
5. Regular JP-4 with dye[a]							
Undersaturation (K-F)	16	---	13	---	17	---	11
Free water (detector)	0	2-3	0	0	0	0	0-1
6. Regular JP-4 with dye[b]							
Undersaturation (K-F)	26	---	12	---	17	---	13
Free water (detector)	0	0	0	0	0	0	0

(a) Added dye to fuel from preceding test; same filter-separator elements used.

(b) New load of fuel and new filter-separator elements.

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	Positive free water	Zero free water
Number of occurrences	3	21
Corresponding undersaturation		
Average	13	21
Range of values	11-16	11-29

Here again, as in the data from the single-element program, the correlation is largely indirect; i.e., there are no instances of extreme discrepancies, but the data are insufficient to conclude that a direct correlation exists between the two methods.

E. Significance of Data on Line Samples

The overall test results using the free water detector on line samples indicate that this test is measuring an entirely different property than that measured by Karl Fischer titrations. Our interpretation of the discrepancies in terms of nonequilibrium conditions (with respect to water saturation) is supported by the higher free water contents shown with direct on-line sampling through detector pads in comparison with the use of bottled samples. Since no positive correlation between the free water detector and the Karl Fischer method is possible under these conditions, acceptance of the free water detector as a valid and reliable method of measuring true free water content must be based mainly on the results of the static sample evaluations, where the use of presaturated fuel samples eliminated or at least minimized deviations from equilibrium conditions. These static evaluations (part reported in Section IV and part in Section VI) indicate that the method is sensitive, reliable, and accurate to within 5 mg/liter (±2.5) or better at free water contents of 0-20 mg/liter.

Assuming that the fairly frequent positive indications of free water in filter-separator effluent streams are real, it is pertinent to consider the significance of these results in terms of field operations. In the case of the single-element filter-separator tests, the element at 20 gpm is probably being stressed at very nearly its maximum capability, and the appearance of occasional small amounts of free water is not too surprising. In the case of the F-6 refueler tests, in which the filter-separator was being tested at only 20-30% of rated fuel flow, the appearance of free water in the effluent fuel was quite unexpected. It now appears at least possible that passage of small amounts of free water into the effluent fuel may be characteristic of many types of filter-separators whenever they are called upon to coalesce and remove substantial amounts of water. This question cannot be resolved until the free water detector has been used extensively both in field operations and in filter-separator and element testing, so as to cover a wide range of equipment and operating conditions.

Free water contents of less than 5 mg/liter, such as were indicated by the detector in this program, may not represent an operational hazard in refueling operations. These amounts are quite small in relation to the amount of dissolved water in the fuel, some of which can in theory separate out as free water in the aircraft fuel tanks during climb and high-altitude flight. Quite possibly the use of "zero free water" as a criterion for filter-separator operation will have to be changed to the use of some acceptably low level of free water, now that an adequate method is available for direct determination of free water content.

SECTION VI

MISCELLANEOUS EVALUATIONS

A.   Evaluation of Field Monitors and Flow Diffusers

Six standard "Millipore MO00 037 PO" field monitors (clear Tenite plastic), each containing a 37-mm uranine-coated test pad, were furnished through RTD for evaluation. The use of a throw-away prepackaged unit of this type would offer considerable advantage in the field. The primary question to be resolved was whether the monitors would seal tightly enough to prevent access of atmospheric moisture and deterioration of the test pads. A plastic flow diffuser was also furnished by the manufacturer; this was a perforated plastic plate fitting inside the monitor, designed to improve the flow pattern and distribute any free water uniformly on the test pad.

The test pads received in the monitors were not suitable for use, as they all showed low-level fluorescence under UV. These pads had not been prepared with the intention of use in quantitative determinations, and apparently they had picked up moisture either during preparation or subsequent storage. Therefore, fresh 37-mm pads were prepared by SwRI and mounted in the monitor housings, after checking the pads for the absence of fluorescence. The monitors were assembled carefully, making sure that the two pieces of the body were tightened securely and that the end plugs were well seated. Duplicate assemblies were stored for 48 hours at room temperature under each of the following conditions: (a) evacuated desiccator, (b) laboratory atmosphere, about 60% relative humidity, and (c) high humidity (sealed chamber with water in bottom). After storage, the pads from these monitors showed, respectively, (a) no fluorescence, (b) low-level fluorescence, and (c) intense fluorescence, more than the 20-mg/liter color standard. This rapid deterioration of the test pads under high-humidity conditions indicated that the sealing of the standard field monitor was inadequate for this purpose. Either supplementary moisture-proof packaging or redesign for tighter sealing would be required to improve this situation.

The flow diffuser supplied by the manufacturer of the field monitors was a 33-mm diameter clear plastic disk, extensively perforated with 0.040" holes. The diffuser was designed to fit snugly into the top half of the monitor to distribute the influent fuel stream over the entire effective filtration area and prevent direct "jet" impingement of the incoming fuel stream on the center of the test pad.

In each evaluation of the flow diffuser, a freshly prepared test pad was mounted with the diffuser in a standard plastic field monitor, the monitor was positioned either horizontally or vertically, and a sample of standard water-in-fuel dispersion was forced through the unit under 10-psi pressure. At this low pressure, it was feasible to use the monitor without any external housing

and to observe the flow visually. It was evident from these observations that the diffuser gave satisfactory distribution of fuel flow. Similar tests were also run under 60-psi pressure, with the field monitor installed inside the "bomb sampler" normally used in line sampling. Control tests were also run using test pads in the laboratory vacuum filtration apparatus. The following results were obtained:

	Standard water-in-fuel dispersion, mg/liter		
	5	10	20
Indicated by detector, mg/liter:			
Monitor vertical, 10 psi	5	10	20
Monitor horizontal, 10 psi	5	8-10	20
Monitor vertical, 60 psi	4-5	10	20
Monitor horizontal, 60 psi	5	10	20
Pad in laboratory filter	4-5	10	19-20

From these data, it is evident that performance of the diffuser was satisfactory in all cases. At the low pressure of 10 psi, the flow diffuser is not essential. However, at 60 psi, if the diffuser is omitted, the flow tends to channel through the center of the test pad. The use of a perforated diffuser without any holes in the immediate center area (about 5-mm diameter) eliminates this channeling.

Based on this limited evaluation, it appears that the use of preassembled disposable field monitors with flow diffusers and test pads would be quite satisfactory if tighter sealing or additional moistureproof packaging can be devised.

For regular use for line sampling in the single-element test program, it was considered more desirable to use the permanent stainless steel type of field monitor such as the "Millipore XX64 037 75." Although this type of monitor is designed for use in the same bomb sampler used for the plastic monitors, the internal dimensions of the stainless steel monitors are such that a different design of flow diffuser must be used with the detector pads. A plastic flow diffuser designed (by SwRI) and constructed for use in the stainless steel monitors came into actual contact with the edges of the test pad, and there was considerable concern that this would block off a substantial portion (about 35%) of the effective flow area. Assuming that the sample size should be reduced proportionately, only 325 ml of fuel would be passed through for each test. However, trials of this assembly indicated that the fuel-wetted area appeared to be essentially as large as that obtained with the plastic monitors; hence, no reduction was made in sample size.

Another factor in this decision was the reasoning that, at the low levels of water contents being encountered in the single-element program, sample

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size in the range from 325 to 500 ml should not give significant differences that can be detected in the color comparison. For example, at a free water content of 2 mg/liter, assuming that a 325-ml sample would give the correct reading with a given flow area of the pad, the use of a 500-ml sample would be expected on the basis of straight proportionality to give a reading of 3 mg/ liter, which is not really distinguishable from the "correct" reading of 2 mg/ liter.

Since this question of sample size had been raised, some experiments were run in the 47-mm laboratory filtration apparatus, comparing 325-ml samples of standard water-in-fuel dispersions with the usual 500-ml samples. The following data were obtained:

	Water content of dispersion, mg/liter		
	5	10	20
Pad rating with 500-ml sample	3-5	9-11	20-22
	4-6	10-12	20-22
Pad rating with 325-ml sample	4-6	10-12	16-18
	3-5	8-10	17-19
Theoretical pad rating with 325-ml sample	3.3	6.5	13

In these tests, there was no significant effect of sample size on the ratings when 5 or 10 mg/liter dispersions were used. With the 20-mg/liter dispersions, the smaller 325-ml samples give slightly lower ratings, but not as low as would be predicted on the basis of straight proportionality. This result was quite unexpected, and thus far no reasonable explanation has been found. However, the data did indicate that sample volume may not be as critical as had been anticipated, and this gave additional support to the use of the partially blocked test pads in the stainless steel monitor with flow diffuser, without change in sample size. However, to eliminate this uncertainty in the future, it will be quite feasible to redesign the flow diffuser to avoid any possibility of test pad edge blocking.

B.   Test Pad Packaging

In order to evaluate the feasibility of packaging test pads in ordinary plastic bags, a supply of 4" X 6" bags of 4-mil polyethylene (Bel-Art Products No. F-2178) was obtained through RTD. These were cut down to half size (4" X 3") for packaging test pads. Fresh lots of 47-mm uranine-coated pads were prepared and checked for original color and the absence of UV fluorescence. Eight of these pads were then placed in individual polyethylene bags, which were closed by heat-sealing. These operations were performed in a

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laboratory environment at about 50% relative humidity. Duplicate bags with
pads were then stored for 100 hours at 70-80°F under different moisture con-
ditions. After storage, the pads were examined for color change and UV
fluorescence and then tested with standard 10 mg/liter water-in-fuel disper-
sion, with the following results:

Storage conditions (100 hr, 70-80°F)	Color (daylight)	Fluorescence (UV)	Pad ratings with 10-mg/liter dispersion	
Vacuum desiccator	Orange (no change)	None	8-10	8-10
Ambient (50% relative humidity)	Slightly yellow	None	8-10	8-10
High humidity (sealed chamber with water)	Slightly yellow	None	10-12	8-10
Submerged in water	Slightly yellow	None	8-10	10-12

Although all of the pads stored under nondesiccated conditions showed
a slight yellowing, there was no indication of UV fluorescence after storage,
and all of the pads gave satisfactory results on the standard dispersion.
Therefore, it appears that packaging in heat-sealed polyethylene bags is ade-
quate to prevent moisture pickup, at least for 100-hour storage at normal
temperature and high humidity.

C.   Evaluation of Commercially Prepared Pads

A limited evaluation was made of pads prepared and individually
packaged by two suppliers, presumably in water vapor barrier material con-
forming to the packaging requirements in the Navy specification for detector
pads, MIL-D-81248(WP). Supplier A furnished 47-mm pads and Supplier B
furnished 37-mm pads. Information from Supplier B indicated that their pads
should not be considered as representative of a quality-controlled production
run; therefore, our evaluation of these 37-mm pads was very limited.

As a comparison standard, 37- and 47-mm pads prepared by SwRI
about two weeks earlier (sealed in polyethylene bags) were included in the
evaluation. It should be noted at this point that we had not been successful in
reducing the dye content of the 37-mm pads in proportion to the decrease in
pad area (compared to 47-mm pads). Most of our 37-mm pads that had been
checked throughout the program for uranine content showed about the same
total amount of dye as did the 47-mm pads. The relatively higher dye con-
centration per unit area in the case of the 37-mm pads did not affect their
accuracy when tested against standard water-in-fuel dispersions. Although
firm quantitative data are lacking, it appears that dye concentrations per unit

area up to about 150% of the desired concentration do not affect the pad sensitivity adversely.

Two 47-mm pads from Supplier A and four SwRI pads (two 37 mm and two 47 mm) were checked for initial quality by removing each pad from its package and immediately observing the pad under ultraviolet illumination. The ambient relative humidity during this operation was 52%. The dye content of each pad was then determined colorimetrically. The following results were obtained:

Source of pads	Dye content, mg	Initial appearance under UV
A, 47 mm	1.48	Slightly whitish, but no fluorescence; usable pad
	1.68	Slighter lighter than zero standard, but good pad
SwRI, 47 mm	0.88	Identical to zero standard
	0.86	Identical to zero standard
SwRI, 37 mm	0.79	Identical to zero standard
	1.06	Identical to zero standard

The dye contents of the pads from Supplier A were quite high. The whitish discoloration observed on one of these pads (under UV) was quite similar to that experienced in earlier SwRI attempts at pad preparation under unfavorable high-humidity conditions. More recent SwRI preparations, as illustrated in the data just cited, gave pads that matched the zero color standard exactly. These data also illustrate the problems encountered in holding down the dye content of the 37-mm pads, since one of the two pads was above even the specification range for 47-mm pads (0.80-0.85 mg). Based on SwRI experience to date in pad preparation, this is an exceedingly narrow range of dye content to meet with a hand spraying operation, and the situation seems to be more difficult with the 37-mm pads. No attempt has been made in this program to develop more reproducible spray techniques suitable for production, but it has become quite evident that such techniques would have to include automatic control of spray schedule and a constant spray pressure, neither of which exist in the hand-spray techniques used here.

Similarly selected sets of six pads were evaluated for accuracy of readings when tested against standard water-in-fuel dispersions (5 and 10 mg/liter), drawing 500 ml of the dispersion through a pad mounted in the laboratory filter holder. Prior to test, each pad was checked for initial appearance under UV. The following data were obtained:

Source of pads	Initial appearance of pad under UV vs zero standard	Water content, mg/liter	
		Standard dispersion	Pad rating
A, 47 mm	Identical	5	5-7
	Identical	5	4-6
	Lighter, still good	10	10-12
	Identical	10	9-11
SwRI, 47 mm	Identical	5	5-7
	Identical	5	5-7
	Identical	10	10-12
	Identical	10	9-11
SwRI, 37 mm	Identical	5	4-5
	Identical	5	5
	Identical	10	8-10
	Identical	10	10-12

All of these results indicated the pads to be of good general quality and accurate in rating level.

Another set of packaged pads was evaluated for quality of packaging, essentially as specified in MIL-D-81248(WP). This set of pads included two each from Suppliers A and B and two SwRI pads. Tightness of packaging was checked first by immersing each packaged pad in a beaker of water and placing in a vacuum chamber. The pressure was lowered to 8.5 in. Hg below atmospheric, and the packages were observed for air leaks, all with negative results. These packages were then stored for 16 hours at 100°F in a sealed chamber with water in the bottom. The condition of the pads after this exposure period was rated as follows:

Source of pads	Appearance of pad under UV
A	No fluorescence; slight fading around edges
	No fluorescence; somewhat whitish
B	No fluorescence, but pad white
	No fluorescence, but pad white
SwRI	No fluorescence; slight fading around edges
	No fluorescence; slight fading around edges

One of the Supplier A pads and both of the SwRI pads would be rated as still good in spite of the slight edge fading. The second Supplier A pad was somewhat marginal, and both Supplier B pads were far enough off color that they would probably be considered unsatisfactory for use. Since no evaluation was

made of any Supplier B pads as received, it cannot be said with certainty when the apparent moisture pickup occurred.

The six pads that had been exposed in the 16-hour humidity test at 100°F were further evaluated on standard water-in-fuel dispersion. All were found to give reasonably accurate results, although the B pads were furthest from the theoretical readings.

Comparing the results on A and SwRI pads after exposure to high humidity with the results cited previously (without such exposure), it appears probable that some water vapor penetrated the packages during exposure of both the Supplier A and the SwRI pads. Exact comparisons cannot be made, since the pretest condition of the humidity tests cannot be determined.

Earlier, when SwRI-prepared pads packaged in polyethylene bags were tested for 100 hours at room temperature over a layer of water in a sealed chamber, there was no evidence whatever of moisture penetration. The slight edge fading observed in the 16-hour tests at 100°F would indicate that these conditions are more severe. This difference could be merely a function of test temperature, or more likely the 100°F tests were made more severe by the relatively rapid temperature rise when the test chamber was first placed in the oven and by the normal oven temperature fluctuations at thermostat cut-in and cutoff points, both of which would be more likely to induce condensation than would the previous room-temperature storage tests. No direct comparison is available between either storage test and that specified in MIL-D-81248(WP), which consists of 16 hours at 100°F in a General Foods type humidity chamber at 95% relative humidity.

D.   Exposure of Test Pads to Fuel

In view of the possibilities of using detector pads in monitor-type housings for "on-stream" field applications, it was of interest to determine whether exposure of the pads to "dry" fuel (containing no free water) would affect their subsequent performance. It was visualized that such exposure might occur inadvertently under field conditions.

Static contact of test pads with fuel was studied by placing an SwRI-prepared 47-mm pad in each of two metal line-type filter holders. Both holders were then filled with test fuel (approximately 40 ml), making sure that all entrapped air was removed, and the holders were sealed tightly with stainless steel caps. The fuel placed in one of the holders represented clean influent fuel taken from the single-element test then in progress; the water content of this fuel was running between 24 and 35 mg/liter, i.e., about half of the saturation value of 65 mg/liter at 75°F. The fuel used to fill the other holder had been equilibrated over water at 90°F and hence was expected to release some free water when cooled to room temperature. Both sealed holders were stored for 16 hours at 76°F, after which the pads were removed and inspected under UV. Both pads were slightly on the maroon side, in

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contrast to the usual purple color of water-free pads under UV, but there was no indication of fluorescence. When these two pads were then tested on standard 10-mg/liter water-in-fuel dispersion, both indicated water contents of 20 mg/liter, i.e., twice the true content.

This result was completely unexpected in the case of the undersaturated influent fuel. Therefore, two fresh pads were checked with this fuel in the same manner, this time storing for 64 hours at 70°F. The results were identical to those of the previous test, indicating that long-term exposure to even relatively small quantities of "dry" fuel under static conditions can cause the pads to give erroneously high results in subsequent use.

It was also considered of interest to check the effect of passage of fairly large amounts of "dry" fuel through the pads. For this purpose, a fresh 47-mm test pad was mounted in a "Gelman 1200C" filter holder, which was then connected to the single-element test loop on the influent side, before the water injection point. "Dry" (undersaturated) influent fuel at 75°F was passed through the unit at approximately 1 gal/hr. Duplicate tests were run with different exposure periods, and each pad was then rated against standard 10-mg/liter water-in-fuel dispersion, with the following results:

| Exposure time, | Pad indication, mg/liter | |
minutes	Test No. 1	Test No. 2
15	10-12	10-12
30	8-10	6-8
60	12-14	6-8

These results indicate that passage of up to a gallon of "dry" fuel (about 50% saturated) through a test pad over a one-hour period does not have any consistent effect on the subsequent performance of the pad. This behavior is in marked contrast to that shown in static exposure where contact with a much smaller volume of similar fuel, but for longer periods, did cause the pads to register consistently high.

Although the flowing-fuel tests did not show any consistent trend, the scatter of results around the "true" value was somewhat greater than that encountered in the normal use of fresh pads. It was considered barely possible that this inconsistency was related to leaching of some of the uranine by the flowing fuel. However, a rough qualitative test on each of the recovered fuel samples failed to indicate the presence of any dye, and significant leaching by fuel appears improbable in view of the predominantly water-soluble nature of the sodium-salt dye.

SECTION VII

POSSIBLE METHODS OF FIELD APPLICATION

In considering the various ways in which the free water detector could be used in quality control of base fuel handling operations, there are three basic possibilities, each with several possible variations:

(1) Fuel system samples could be collected in bottles and taken to the laboratory for analysis using the detector pads in any convenient filter holder.

(2) Test pads could be mounted by the laboratory in permanent or disposable field monitors designed for direct field sampling through the pad; after passing the required fuel volume through the assembly, it would be taken back to the laboratory for checking the pad in the detector box.

(3) Preassembled disposable type field monitors (each containing a test pad) could be purchased, and the rating of the pads could be accomplished in a detector box at or near the sampling site.

The first of these possibilities, involving the use of sample bottles, does not appear attractive under most Air Force base conditions. Since sampling sites are in many cases far removed from the laboratory, there would inevitably be a delay of at least several minutes and possibly up to an hour between sampling and testing. During this time, many things could go wrong with the sample. Temperature changes could cause dissolving and disappearance of free water originally present, or the liberation of water originally dissolved; for a fuel with a steep saturation/temperature curve, temperature changes of only 10°F could introduce errors of 10 mg/liter or more. There is also the problem of settling out of free water, which may be extremely difficult to redisperse properly for analysis. Finally, even if temperature changes and water settling can be eliminated or taken into account, there is still the very real problem represented by "nonequilibrated" samples. Although such conditions are believed to be much more of a rarity in field operations than in filter-separator testing, there is some evidence that they can occur in normal base fuel handling. The rate of disappearance of free water under nonequilibrium conditions can be quite rapid in terms of the times required to transport bottled samples from refueling sites to the laboratory. The work reported herein had demonstrated that as much as 5 mg/liter of free water can disappear in about a minute, and it is reasonable to extrapolate this disappearance rate upward to predict the disappearance of as much as 20 mg/liter of free water, given a sufficiently undersaturated fuel, a relatively fine dispersion of the water, and a waiting time on the order of 30 minutes. It may be argued that the presence of free water in such a sample at the time it is drawn is not significant, since the same sort of redissolving will presumably occur in the aircraft tanks after refueling is completed. However,

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the passage of free water in any substantial quantities is a definite indication of filter-separator malfunctioning, and such a unit would be just as likely to pass free water along with fully saturated fuel; such equipment should be deadlined.

In view of these circumstances, the use of bottled samples with the free water detector in base fuel quality control appears very unattractive, except as a last resort. Possibly the problems involved are not as serious as we have indicated, but this would have to be demonstrated by extensive field trials or by additional test-loop work aimed directly at resolving this question. The line-sample free water data of the program reported herein were somewhat incidental to the main purpose of the program, and hence tend to point out possible problem areas in the use of the apparatus without providing a definitive solution.

For a decision between the other two possibilities listed, or variations thereof, the relative economics will have to be taken into account, as well as purely technical considerations. Since the ultimate prices of detector boxes, test pads, and throwaway type field monitors have not yet been established by experience, this report will discuss primarily the technical considerations involved.

First of all, it should be noted that the detector box covered by the present specification is not suitable for most on-site applications, as it requires a 120-volt ac outlet and is not built for use in hazardous-vapor areas. Modification of the circuitry to permit operation from 12-volt dc would be fairly simple and inexpensive, but modification to meet explosion-hazard requirements for use in the field would very likely involve considerable redesign and added cost, whether vapor-tight or full explosionproof design were required.

If a detector box suitable for on-site use is feasible economically, then there does not appear to be any other absolute barrier to its successful use by refueling personnel. The actual operations of passing the sample through the monitor unit and then comparison-rating the test pad are simple enough to be accomplished by relatively unskilled personnel when proper instructions are provided. The only auxiliary piece of equipment that would be required is a hand-operated suction pump, syringe, or rubber suction bulb for removing the excess fuel from the test pad. Perhaps the most serious problem to be resolved would be how to perform the various operations during periods of heavy rain or snow without getting extraneous water onto the test pad, which could occur during connecting or disconnecting the monitor as well as in the subsequent disassembly and rating. The latter operations, in the case of truck or semitrailer refueling, could be performed within the cab of the truck or tractor. In the case of hydrant refueling, hose carts such as the MH-2 are towed to the aircraft by a pickup truck or other vehicle and are sometimes left at the refueling site while the truck proceeds elsewhere. In

such a case, it might be necessary to locate the detector box in the operating storage pumphouse, or in the pump control room if the pump area itself is the open-shed type.

Although rating the test pads at or near the refueling site would present definite problems, it appears that these could be resolved successfully in order to take advantage of the "instant ratings" that could be obtained.

With regard to the type of test pad holder to be used in the sampling, it appears that this selection will depend mainly on the success of the equipment manufacturers in developing a satisfactory low-price throwaway type of housing that will keep the test pad in good condition from the time of manufacture to the time of use. If such an assembly is not developed, then the final installation of the test pad would have to be performed shortly before use; this could be accomplished best in the laboratory. In either case, the housings would in all probability be designed to fit into the "bomb sampling kit" that has already been furnished to many Air Force bases for determination of particulate matter.

It should also be noted that the simplicity and rapidity of free water determinations using the detector may well permit its use on samples other than those taken during refueling operations. For example, it could be used to check incoming fuel, fuel from bulk and operating storage tanks, and fuel dispensed to refueling vehicles. Some of these samples could be obtained as line samples, in the same manner as the refueling samples would be obtained. Direct sampling from tanks might also be necessary (e. g., incoming tank cars or tank trucks, bulk storage tanks, operating storage tanks, and refueler tanks). In most of these instances, not enough fuel head would be available to force the sample through the water detector pad at a reasonable rate, and the use of an inexpensive hand suction pump would be necessary.

Any detailed recommendations on points and frequency of sampling are beyond the scope of this report. The important fact to be noted is that the free water detector tests are sufficiently simple and rapid to broaden the possibilities of quality control throughout the base fuel handling system.

Apart from the use of the free water detector in fuel quality control at the base level, it appears to be entirely suitable for use as the primary method of water detection in the preproduction testing of filter-separators and elements.

SECTION VIII

SUMMARY AND CONCLUSIONS

The free water detector provides a rapid method for the quantitative detection of free water in jet fuel. It is simple in operation and should be usable even by relatively unskilled personnel. In its present form, it is designed for use in the laboratory but could be modified for use in the field.

Based on the data reported herein, the repeatability of the results is well within 5 mg/liter (±2.5 mg/liter) in the range of free water contents up to 20 mg/liter. In the lower end of this range, up to 10 mg/liter, closer checks can be obtained by interpolation, but these are somewhat dependent on the skill and color vision of the operator rating the pads.

The accuracy of the method (as distinguished from its precision) is somewhat more difficult to assess, since the Karl Fischer ratings used for comparison in most of the work reported herein cannot be used as absolute standards. In static evaluations of the detector using standard dispersions of free water in saturated fuel, no deviations of more than 3 mg/liter were noted between the amount of water added and the indicated value, when using correctly prepared test pads in the normal manner.

When applied to fuel line samples taken during handling tests, the free water detector often indicates the presence of traces of free water even when the Karl Fischer data show values of total water content well below saturation. It is believed that these detector indications of free water content are valid and that the detector ratings are more significant than the Karl Fischer results as a measure of line-sample quality. For samples on which the Karl Fischer results indicated undersaturation greater than its estimated repeatability, the detector never indicated more than a trace of free water (less than 1 mg/liter). In the single instance in which free water in excess of 10 mg/liter appeared in the fuel effluent stream, the detector and Karl Fischer results were in excellent agreement.

No systematic investigation was made of the possible effects of fuel additives and contaminants on the performance of the free water detector. However, tests involving the presence of 0.08% anti-icing additive and an unknown amount of corrosion inhibitor did not show any unexpected effects due to the presence of these additives. Also, a yellow dye proposed for addition to JP-4 fuel in amounts of 1 lb/1000 gal was found to have no effect on performance of the detector. Contents of particulate matter (primarily red iron oxide) up to 0.9 mg/liter apparently had no adverse effect.

It should be appreciated that most of the data reported here was obtained in conjunction with test programs aimed at objectives other than evaluation of the detector; hence, no systematic comparison of detector readings and Karl

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Fischer values was made under controlled conditions of true water content, amounts of various additives, and amounts of different types of particulate matter.

The free water detector in its present form is far superior to the Karl Fischer method in ease of performance and is believed to give more reliable and meaningful results. Successful application of the free water detector as a field quality control method may be somewhat dependent on the development of modified equipment for field use, in particular preassembled throwaway field monitors and a detector box of either vapor-tight or explosionproof construction.

SECTION IX

RECOMMENDATIONS

It is recommended that some additional static evaluations should be performed with the free water detector using fuel and water phases containing additives and/or contaminants commonly encountered in the field. In particular, it is recommended that these studies should include the effects of pH of the water, which can vary over rather wide limits under field conditions. Since the dye is present on the pads in the form of the water-soluble sodium salt, it is possible that acidic water phases will not give the same fluorescence as that obtained with essentially neutral water. These studies should also include the use of free water containing various concentrations of anti-icing additive, which may be as high as 40% under winter conditions in the field, and various amounts of sea water contamination. Further studies of the effects of fuel additives and solid contaminants should be made. It is possible that some of these recommended studies have already been performed by the Navy but not yet published, so that the recommended program can be reduced if sufficient information becomes available along these lines.

Continued use of the free water detector is recommended for the evaluation and development programs presently being conducted for the Air Force by SwRI, with a reduction in frequency of the Karl Fischer analyses that are performed.

It is also recommended that any commercially prepared test pads from new sources and any new types of pad holders designed for field use should be evaluated.

Field trials of the free water detector in its present form are recommended, both in fuel quality control operations and in preproduction testing of filter-separators and elements. At the same time, it is recommended that a feasibility study should be made on the modification of the present unit for battery-powered operation in hazardous-vapor areas.

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13. ABSTRACT

A test apparatus and procedure for detecting small amounts of free water in jet fuels have been evaluated. A fuel sample is passed through a dye-coated pad, which is then rated for fluorescence under ultraviolet illumination in comparison with color standards. Based on static evaluation of standard samples, the detector has accuracy and precision well within 5 mg/liter on samples containing up to 20 mg/liter free water. The detector often indicates traces of free water in filter-separator effluent samples for which Karl Fischer analyses indicate undersaturated fuel, which is explained by the nonequilibrated state of such samples. Normal concentrations of anti-icing additive, corrosion inhibitor, or iron oxide contaminant do not affect the sensitivity of the detector, based on limited data. The detector is far simpler and faster than the Karl Fischer method and is believed to give more meaningful results. It appears to be very suitable for use in preproduction tests of filter-separators and elements. The usefulness of the detector in base fuel quality control will depend somewhat on availability of suitable prepackaged pad holders and viewing comparators. Direct line sampling through a detector pad is more reliable than the use of bottled samples. Field trials and further laboratory evaluations are recommended.

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