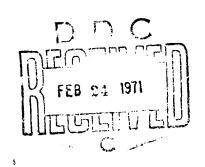
EVALUATION OF THE AQUA-GLO SERIES II FREE WATER DETECTOR

CHARLES R. MARTEL

AIR FORCE AERO PROPULSION LABORATORY

TECHNICAL REPORT AFAPL-TR-70-63

NOVEMBER 1970



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FOREWORD

This report was prepared by the Fuel Branch of the Fuel, Lubrication, and Hazards Division, Air Force Aero Propulsion Laboratory, and is documented under Project 3048, Task 304805. The work was performed inhouse by Air Force personnel using facilities of the Air Force Aero Propulsion Laboratory. The Air Force project engineer for this program was Mr. Charles R. Martel (APFF). This report was submitted by the author 11 August 1970.

This program was initiated to determine the suitability of the Aqua-Glo Series II for measuring the undissolved water content of fuel during acceptance testing of filter/separators and for field use by the Air Force. This program will also provide the American Society for Testing and Materials Panel on Free Water in Fuel Analysis with information that may be useful in selecting a standard method for analysis of free (undissolved) water in hydrocarbon fuels.

The author wishes to express his appreciation to Mr. Carl R. Hoke of the Air Force Aero Propulsion Laboratory (APMT) who performed the tests and collected the data necessary for this program.

This technical report has been reviewed and is approved.

ARTHUR V. CHURCHILL

Chief, Fuel Branch

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ABSTRACT

The Aqua-Glo Series II free water detector, for use in measuring the quantity of free (undissolved) water in hydrocarbon fuels, was evaluated. Two dynamic test loops and a batch-blending calibration apparatus were used to check the accuracy and repeatability of the Aqua-Glo Series II. The Aqua-Glo's response to free water in fuel was found to be affected by the presence of fuel additives in the fuel and by the degree to which the water was dispersed in the fuel. Corrosion inhibitors such as Santolene C, AFA-1, and Lubrizol 541 were found to decrease the Aqua-Glo's response to free water while the fuel system icing inhibitor was found to increase the Aqua-Glo's response to free water. A poor dispersion of the free water in fuel, i.e., one that has a few large drops versus a good dispersion, where there are many small drops, resulted in decreased repeatability and a lowered Aqua-Glo response.

In general, the Aqua-Glo Series II free water detector was found to be suitable for field use in its present configuration over the range of 2 to 40 milligrams per liter of water in the fuel. For laboratory use, the accuracy can be maintained within ±10% if the Aqua-Glo is first calibrated for use with each particular fuel/fuel-additive blend. The Aqua-Glo Series II is superior to the AEL free water detector in terms of accuracy, repeatability, and portability.

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SECTION I

INTRODUCTION

1. GENERAL

The objective of this in-house program was to determine the accuracy of the Aqua-Glo Series II free water detector, manufactured by the Gammon Technical Products, Inc., Newark, N. J., and to compare it to the Navy's Aeronautical Engine Laboratory (AEL) free water detector. Both 25-mm and 37-mm pads were used, and possible sources of error were examined.

The detection and measurement of free water in hydrocarbon fuels, especially aviation jet fuels, has been a major problem for the Air Force and other agencies for many years. Free (i.e., undissolved) water in jet fuels presents a serious safety hazard to aircraft as the water may freeze into ice crystals and plug fuel filters leading to engine flameout during flight. In addition, free water accelerates corrosion and the growth of microorganisms in aircraft fuel tanks.

2. BACKGROU'ID

A primary Air Force need for an accurate free water detector is for testing fuel filter-separators. Specification MiL-F-8901B requires that a filter-separator pass no more than 5 milligrams per liter (mg/l) free water. The measurement of the free water and the accuracy of the measuring technique have always created problems during filter-separator qualification and testing.

a. Karl Fisher Titration Method

For many years the Karl Fisher titration method was used to measure the water content of hydrocarbon fuels. To determine free water content, however, it was assumed that the fuel was water saturated and the difference between the measured value and the water-saturated value was the free water content. There is evidence to suggest that this assumption is not always correct. Other shortcomings with the Karl Fisher titration method included: (1) minimum error of about 6 mg/l, (2) sampling errors since a substantial time would usually elapse between sampling and analysis, (3) procedure errors in performing the Karl Fisher titration, and (4) possibly intentional errors because a skilled operator could easily alter the results obtained. See Reference 1.

b. AEL Free Water Detection

About six years ago, the U.S. Navy's Aeronautical Engine Laboratory at Philadelphia, Pennsylvania, developed a free water detection method, commonly referred to as the Art method. Sodium fluorescein dye is applied to one side of an absorbent filter pad, and any subsequent contact of the dye with free water will result in a bright yellow fluorescene when the dye is illuminated by untraviolet light. To use the AEL free water detector, the operator passes a 500-ml fuel sample through the treated filter pad and then compares the pad with standards while it is illuminated under ultraviolet light. Visual comparison of the pad with four standards (rated at 0, 5, 10, and 20 PPM) enable the operator to rate the pad to within about 3 PPM.

Subsequent work with the AEL method revealed the importance of using only line sampling and the problems of calibration (Reference 2). One of the inaccuracies associated with the AEL method is the visual comparison of the pad with the standards. The degree of water dispersion in the fuel directly affects the fluorescence pattern on the pad. If the pad fluorescence pattern does not closely match the fluorescence pattern of the Standards, a considerable error is likely. The human eye does not appear to be an accurate device for averaging a few bright spots over a given area.

The AEL device is presently in use within the U.S. Navy and Air Force, and specifications are available covering the standards (MIL-S-81282), the viewing box (MIL-V-81227), and the pads (MIL-D-81248). Gammon Technical Products, Inc., has marketed a similar device called the Aqua-Glo; also the Millipore Filter Company has marketed a similar device called the Hydro-Scan. Although there are minor differences in pad size, type of standard used, and means of comparison, the same basic technique is used (i.e., visual comparison of the pad with a standard).

c. Aqua-Glo Series II Free Water Detector

In early 1969, Gammon Technical Products, Inc., introduced the Aqua-Glo Series II, which is basically the original Aqua-Glo with a photocell comparator to eliminate the need for visual comparison of the pad with the standard. Also in 1969 the Americal Society for Testing and Materials (ASTM) formed a new panel to develop a proposed test method for measuring small quantities of free water in Juels. The new Aqua-Glo Series II was selected as a promising device worthy of evaluation.

SECTION II

TEST EQUIPMENT AND PROCEDURES

Under a recent Air Force contract, Southwest Research Institute conducted a program to determine the accuracy of the AEL Free Water Detector (Reference 2). The calibration apparatus used consisted of a batch-blending process using a Waring blender to emulsify a known volume of water in a known volume of fuel. Initial test results obtained with a similar apparatus gave poor repeatability and results, so an alternate method was devised and is described below.

1. CALIBRATION APPARATUS

Since there is no accepted calibration technique or standard for use in checking the accuracy of the Aqua-Glo Series II, a special calibration apparatus was designed. One of the primary design goals was to avoid prolonged contact of the injected water with the fuel so as to prevent the dissolution of the water into the fuel. Also, the apparatus was designed to provide a minimum surface area between the water injection point and the Aqua-Glo pad to prevent the injected water from adhering to or being absorbed by the equipment walls. These objectives are accomplished by the injection of the water into the fuel immediately before it passes through the Aqua-Glo test rad. The injected water is dispersed into the fuel by turbulence at the water injection point. Figure 1 shows the schematic diagram of the calibration apparatus, and Figure 2 shows the actual device.

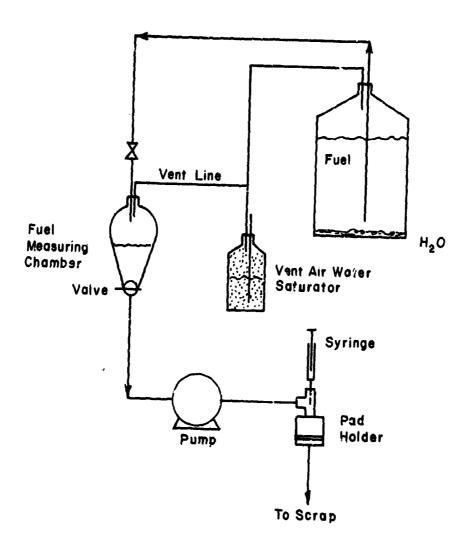


Figure 1. Schematic of Calibration Apparatus

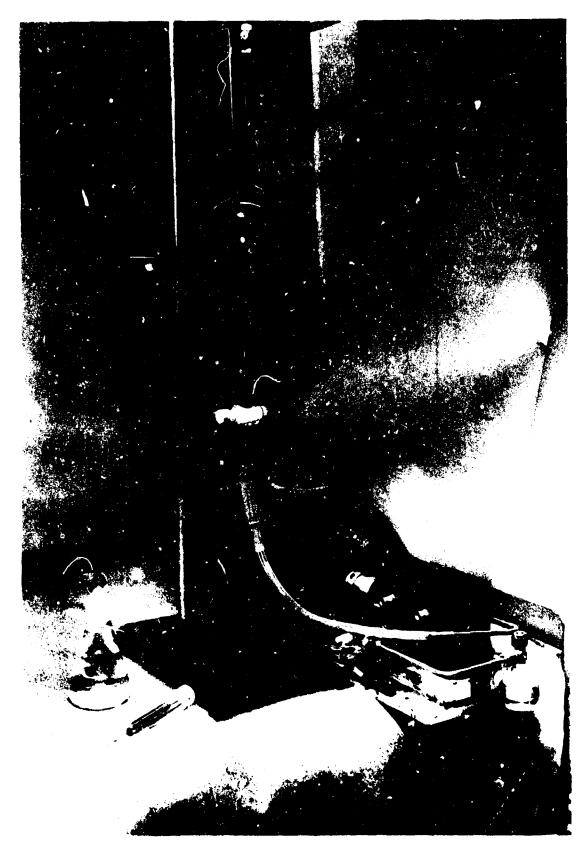


Figure 2. Calibration Apparatus

In operation, fuel is stored in a 5-gallon glass jug with a small quantity of distilled water in the bottom of the jug to allow the fuel to become water saturated. A separatory funnel serves both as a fuel quantity measuring chamber and as the pump feed chamber. The positive displacement pump forces the fuel through the test pad at a flow rate of about 1 liter/minute. The pump effluent passes through 1/4-inch stainless steel tubing to a 1/4-inch tee and then into the pad holder and through the test pad. The top leg of the tee is fitted with a rubber septum through which a hypodermic syringe needle is inserted for injecting the water. The 1/4-inch tubing from the pump to the tee is fitted with an off-center orifice of 0.060-inch diameter where it enters the tee. This orifice was made by filling the tubing end with silver solder, and by drilling an 0.060-inch diameter hole. The off-center orifice creates sufficient turbulence within the tee to emulsify the water before it reaches the test pad. A closed vent system is provided to allow only water-saturated air to enter the system to maintain the fuel in a watersaturated condition.

The equipment used in the construction and operation of the calibration apparatus consisted of the following:

Pump - Positive displacement pump; Northern Ordnance Division of the FMC Corporation; Size 434702A72-5; Serial Number 4979; equipped with a Graham variable speed drive and driven by a 1/4 horsepower; 3450-RPM electric motor.

Hypodermic Syringe - Hamilton Company; 0 to 10 microliter; Model No. 701.

Pad Holders - 25 mm; Gammon Technical Products, Inc.; Part No. GTP-191 37 mm; Millipore Filter Corporation Separatory Funnel - 1000-ml size calibrated and marked at levels of 100, 200, and 500 ml.

In operation, the calibration apparatus worked quite well. The injected water was well dispersed and the results were repeatable. It should be noted, however, that this apparatus does not give a homogeneous water/fuel mixture -- rather the water is injected into the fuel after it starts to flow through the pad, and the injection of the water is completed before all of the fuel sample has passed through the pad.

The test procedure developed and used with the calibration apparatus is as follows:

- a. Siphon a predetermined quantity of fuel from the storage jug into the separatory funnel.
- b. Place an unused Aqua-Glo (25-mm) or AEL (37-mm) pad in the appropriate pad holder.
- c. Fill the microliter syringe with distilled water and expel all but the quantity of water desired. Position the microliter syringe above the pad holder with the needle extending about 3/4 inch through the septum and into the tee. Utmost care must be taken to prevent the inadvertent injection of water from the syringe during this operation. A clamp was used to hold the syringe in position. Recheck the quantity of vater in the syringe after it is in position and before proceeding.
 - d. Open the valve between the separatory funnel and the pump.
- e. Turn on the pump, and, after steady fuel flow is obtained (usually 2 to 3 seconds), slowly inject the water from the syringe into the flowing fuel stream. The water injection must be completed before the fuel flow ceases.

- f. After all the fuel in the separatory funnel has been pumped through the pad, turn off the pump.
- g. Carefully remove the test pad using tweezers, blot well four times, place in the Aqua-Glo Series II instrument, and read. A stopwatch was used to measure the time from start of fuel flow through the test pad until the Aqua-Glo reading is made. A standard time of 3 to 3-1/4 minutes was used.
- h. The ultraviolet light in the Aqua-Glo should be turned off when not in use to conserve battery power. The electrical photocell readout system in the Aqua-Glo Series II has its own separate 9-volt DC battery, and approximately 60 seconds is required for this system to stabilize each time it is used. This 60 seconds is included in the 3 to 3-1/4 minutes time referenced above as the standard reading time. Also, the Aqua-Glo Series II was calibrated before use each day per Gammon Technical Products instructions.

2. DYNAMIC TEST LOOPS

Two test loops (600-GPM and 15- to 60-GPM) were also used to check the calibration and operation of the Aqua-Glo Series II free water detector. Both test loops are installed in Building 42D, Area B, Wright-Patterson AFB, Ohio. Fuel storage for the 600-GrM test loop consisted of a 15,000-gallon underground tank located approximately 80 feet from the test area. The tank is equipped with a 600-GPM deepwell turbine pump and contained about 12,000 gallons of fuel. Underground lines connect the tank with the test area. Figure 3 shows the schematic of a portion of the 600-GPM test loop including the 7-GPM centrifugal pump used to disperse the water in the fuel. Before the

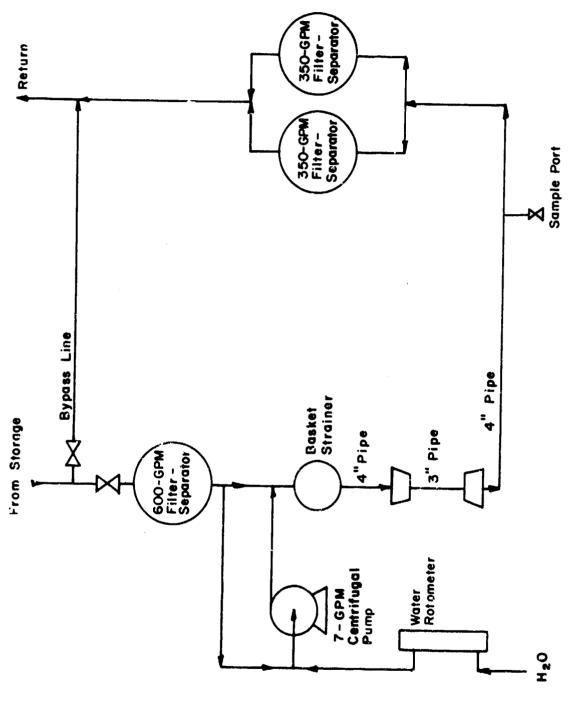


Figure 3. Schematic of 600-GPM Test Loop

7-GPM centrifugal water dispersal pump was added to the system, the degree of water dispersion was found to vary with fuel flow rate and affected the results obtained.

The small test loop is a 15- to 60-GPM system and is well documented in Reference 3. Only 600 gallons of fuel was used in the small loop tests. Fuel flow was measured using a totalizing flow meter and stopwatch, and water flow was measured using a rotometer with a useful range of 0.4 to 1.2 milliliters/minute. Fuel flow rate was maintained at 38 GPM through the tests. The injected water was dispersed into the fuel using an in-line 100-mesh screen with an area of 2.2 square inches.

No attempt was made to presaturate the fuel with water in either loop, but no evidence of loss of water by dissolution into the fuel was found. The fuel flow was measured using a Brodie totalizing flow meter and stopwatch, and the water injection rate was measured using a rotometer. Fuel samples were taken directly from the flowing stream through the test pads using standard Gammon Technical Products, Inc., and Millipore Corporation line sampling equipment.

The test procedures used with both the large and small test loops are similar and consist of the following:

- a. Manually adjust the fuel flow rate to that desired (between 150 and 600 GPM on the large loop and 38 GPM on the small loop).
- b. Manually adjust the water injection rate to that desired. Allow a minimum of 10 minutes of operation at each new setting to allow the system to stabilize.

- c. After achieving stable operating conditions, take in-line samples of the fuel through the 25-mm Aqua-Glo pad or the 37-mm AEL pad. Aqua-Glo Instrument No. 1 (described in Section II-3) was used to rate each Aqua-Glo pad 2-1/2 to 3-1/2 minutes after sampling was initiated. Sample sizes varied depending upon the quantity of water injected in the fuel. The standard sample size for the Aqua-Glo Series II was 500 ml but small sample sizes were used whenever the fuel free water content exceeded the range of the Aqua-Glo. Sample size corrections were then made by multiplying the Aqua-Glo reading by 500 and dividing by the actual sample size (in milliliters).
- d. A minimum of two samples was taken at each fuel/water flow condition at 5-minute intervals. Checks with zero-injected water were made periodically to insure that no free water, other than that purposely injected, was present in the fuel.
- e. With the small loop, three continuous fuel quality monitors were also used—a Bowser Totamitor located upstream of the water injection point and a Bowser Totamitor and a Keene Turbidimeter located downstream of the water injection point and quite close to the sampling point. These monitors permitted a continuous check on the operation of the system and also permitted a direct comparison of their accuracy with that of the Aqua-Glo Series II.

3. AQUA-GLO SERIES II INSTRUMENTS

The Aqua-Glo readout is in PPM (parts per million by volume of undissolved water to fuel). However, in this report the Aqua-Glo Series II readout is listed in mg/l (milligrams of water per liter of fuel). This is numerically equal (to within 1/2%) to parts per million by volume

at the ambient temperatures encountered during the tests (one gram of water occupies one milliliter of volume at 4° C).

The Aqua-Glo Series II instruments used were Aqua-Glo Series!
Instruments converted to the Series II configuration using modification kits obtained from Gammon Technical Products, Inc. Instrument No. 1 has the red painted hinged flap and was modified using Conversion Kit GTP-324B.
Instrument No. 2 has the nickel-plated hinged flap and was converted using Conversion Kit GTP-324A. The conversion kits consisted of a new ultraviolet bulb, an additional light filter, a new standard for permanent installation within the instrument, a calibration standard for periodic use, and a photocell detector assembly which replaces the visual viewing hood. Figures 4 and 5 show front and back views of Instrument No. 1.

One slight difference between the two conversion kits was the padcentering plate installed with a pressure-sensitive adhesive in Instrument No. 1. This thin plate has a hole of about 26-mm diameter to aid in centering the Aqua-Glo pad with respect to the hole in the bottom of the Aqua-Glo. Instrument No. 2 did not have this pad centering device and it was greatly missed. However, for viewing 37-mm AEL pads, the centering device was of no value. Ideally two-pad centering plates should be provided--one for use with 25-mm pads and one for 37-mm pads. The user could then install whichever one he would normally use.

Each Aqua-Glo instrument was also equipped with a carrying case, a rechargeable nickel-cadmium battery, a separate battery charger, a sampling probe and pad holder, and a pair of tweezers.

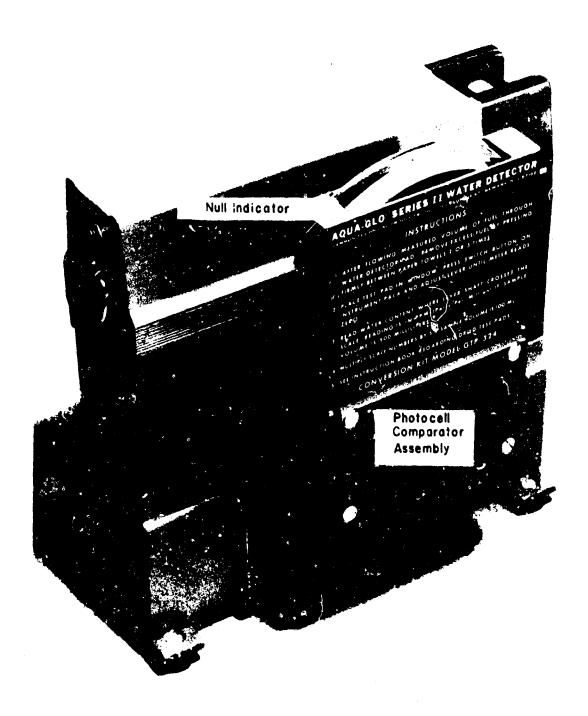


Figure 4. Front View of Aqua-Glo Series II Instrument No. 1

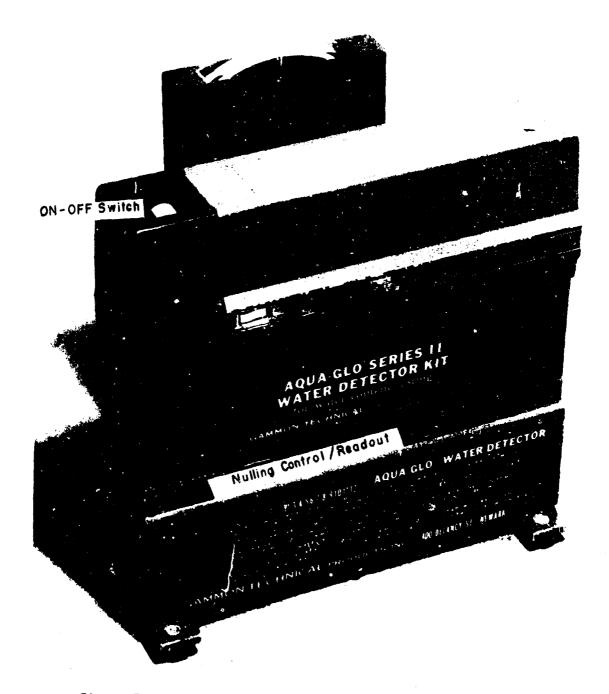


Figure 5. Back View of Aqua-Glo Series 1: Instrument No. 1

Suggested improvements in the Aqua-Glo Series II instruments include:

- i. incorporation of the nickel-cadmium battery charger into the basic instrument thereby eliminating one item of equipment and the need for periodic removal and replacement of the nickel-cadmium battery for recharging.
- 2. Improved pad holder. The 25-mm pad holder design is deficient as the pad usually remains in the upper part of the holder when the bottom portion is unscrewed. This may result in the pad being damaged or dropped while trying to extract it. Also, the water dispersal pattern on the pad was not uniform; the center of the pad received less water than the rest of the pad.
- 3. Redesign of the Aqua-Glo so that it would be more stable while inverted for installation of the pad.
- 4. The incorporation of a small pilot light to glow whenever the ultraviolet bulb is on. This would remind the operator to turn off the bulb when it is not in use to conserve battery power and also to show whether or not the ultraviolet bulb is on. An alternate approach would be the use of a single push button to turn on both the ultraviolet bulb and the photocell readout assembly. Thus, whenever the push button would be released, both circuits would aucomatically be turned off.
- 5. The bottom portion of the pad holder should be provided with means for attaching a graduated plastic bottle. This would aid in collecting and measuring the quantity of fuel passed through the pad.
- 6. A flush bypass valve should be provided on the inlet to the pad holder to permit the sample line to be flushed prior to use.

4. AQUA-GLO AND AEL TEST PADS

Aqua-Glo and AEL test pads are identical except for size. The pads are paper prefilter discs coated with a sodium fluorescein dye. The 25-mm pads used were obtained from Gammon Technical Products, Inc., and the 37-mm AEL pads were obtained from Air Force Stock, Stock Number 6630-105-4386, manufactured by Driaire Inc., E. Norwalk, Connecticut.

5. TEST FUELS AND FUEL ADDITIVES

All tests were run on neat JP-5 fuel conforming to Specification MIL-J-5624 unless otherwise noted. The JP-5, as received, contained no corrosion inhibitor additives and no fuel system icing inhibitor additive.

A limited number of tests were performed using JP-4 fuel that fluoresced when illuminated with ultraviolet light. This fuel also conformed to MIL-J-5624 but it did contain undetermined quantities of fuel additives including a corrosion inhibitor and fuel system icing inhibitor.

The following fuel additives were used for specific tests:

Santolene C, Edwin Cooper, Inc. (formerly manufactured and marketed by the Monsanto Chemical Company). A corrosion inhibitor qualified to Specification MIL-1-25017 and listed under QPL-25017-7. Normal use limits range from 4 to 16 lbs/1000 bbls concentration.

AFA-1, E. I. DuPont de Nemours Co. A corrosion inhibitor qualified to Specification MIL-1-25017 and listed on QPL-25017-7. Normal use limits range from 4 to 16 lbs/1000 bbls concentration.

Lubrizol 541, The Lubrizol Corporation. A corrosion inhibitor qualified to MIL-1-25017 and listed on QPL-25017-7. Normal use limits range from 5 to 20 lbs/1000 bbls.

Fuel System Icing Inhibitor (FSII), conforming to Specification
MIL-I-27686 and composed of 99.6% by weight of ethylene glycol monomethyl
ether and 0.4% by weight of glycerol.

6. DATA ANALYSIS

Straight line relationships were assumed for the Aqua-Glo Series II and the AEL ratings versus free water injected. Where applicable, a linear regression curve (the best fitting straight line for a set of data points based on the method of least squares) was calculated, where X is the milligrams per liter of free water injected and Y is the Aqua-Glo or AEL reading in milligrams per liter. Also, for each regression curve, the correlation coefficient (r) was calculated. The correlation coefficient is a measure of the degree of scatter of the data points in regard to the regression curve. A correlation coefficient of zero would indicate no correlation whatsoever, and a correlation coefficient of -1 or +1 would indicate perfect correlation; i.e., all data points falling exactly on the regression curve.

The equations used to calculate the linear regression curve coefficients for the curves Y = A + BX and X = C + DY are as follows:

$$C = \frac{N\Sigma\lambda_{s} - (\Sigma\lambda)(\Sigma\lambda_{s})}{N\Sigma\lambda_{s} - (\Sigma\lambda)_{s}}$$

$$D = \frac{N\Sigma\lambda_{s} - (\Sigma\lambda)(\Sigma\lambda_{s})}{N\Sigma\lambda_{s} - (\Sigma\lambda)_{s}}$$

$$D = \frac{N\Sigma\lambda_{s} - (\Sigma\lambda)(\Sigma\lambda_{s})}{N\Sigma\lambda_{s} - (\Sigma\lambda)(\Sigma\lambda_{s})}$$

where

$$\Sigma X = \sum_{i=1}^{N} X_i, \quad \Sigma Y = \sum_{i=1}^{N} Y_i, \quad \Sigma X^2 = \sum_{i=1}^{N} X_i^2$$

$$\Sigma Y^2 = \sum_{i=1}^{N} Y_i^2, \quad \Sigma XY = \sum_{i=1}^{N} X_i Y_i,$$

and N = number of data points.

The correlation coefficient (r) is calculated using the following equation:

$$V = \frac{N\Sigma XY - (\Sigma X)(\Sigma Y)}{\sqrt{\left[N\Sigma X^2 - (\Sigma X)^2\right]\left[N\Sigma Y^2 - (\Sigma Y)^2\right]}}$$

SECTION III

TEST RESULTS AND DISCUSSION

1. AQUA-GLO SERIES II RESULTS USING THE CALIBRATION APPARATUS

Several series of tests were conducted using the calibration apparatus with neat JP-5 with sample sizes of 500 and 100 ml, and JP-5 containing various fuel additives with sample sizes of 500 ml.

a. 500-ml Samples of Neat JP-5

Two separate test series were conducted using neat JP-5 and sample sizes of 500 ml. Figure 6 gives the regression curves for these two series and the regression curve obtained using both sets of data. There is a significant different between the two test series. The cause of this difference has not been identified but may be due to one batch of fuel being contaminated with a fuel additive or surfactant. The regression curve obtained with the first test series (Y = -0.13 + 1.55X) was used as the standard for comparison for subsequent tests, since it is based on the most number of data points and is in excellent agreement with subsequent runs on neat JP-5 using 100-ml fuel samples.

Figure 7 shows plots of the Y on X regression curve and the X on Y regression curve for the first test series. The Y on X curves are of value for comparing subsequent tests, but the X on Y curve should be used when converting Aqua-Glo readings to actual free water content. However, with such a high correlation coefficient (r), there is little difference between the Y on X and the X on Y curves.

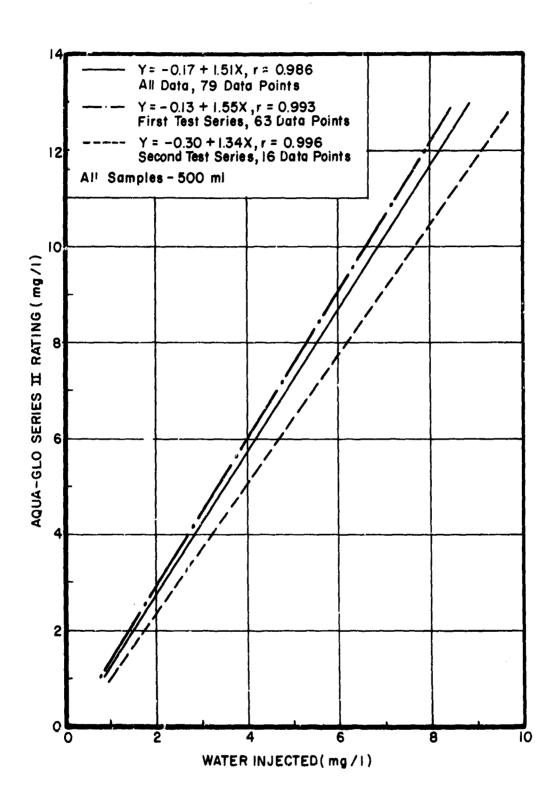


Figure 6. Aqua-Glo Response With JP-5 Fuel Using Calibration Apparatus

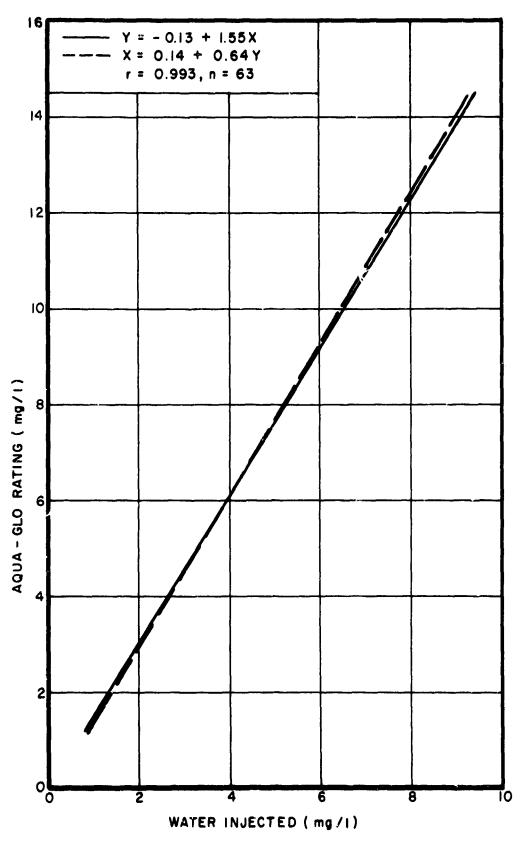


Figure 7. Aqua-Glo Regression Curves for JP-5 Fuel Using Calibration Apparatus

When the X on Y regression curve is used, a correction factor must be applied to the Aqua-Gio readings. If an Aqua-Gio reading of 10.0 were obtained, the corrected free water content would be X = 0.14 + 0.64(10) = 6.54 or about 6.5 mg/l. Thus, the Aqua-Gio Series II reads about 50% too high on neat JP-5.

Note that, in calculating the regression curves from the Aqua-Glo data, all points at zero injected water are neglected inasmuch as the Aqua-Glo does not read below 1 mg/1; i.e., it is impossible to read "zero" on the Aqua-Glo as the scale does not extend below 1 mg/1. Plotting of actual Aqua-Glo data points versus actual free water injected always resulted in a sharp curve at the bottom of the linear response curve indicating that below about 1-1/2 to 2 mg/1 the Aqua-Glo response is non-linear.

b. 100-ml Samples of Neat JP-5

When the calibration apparatus is used, the water is mixed with the fuel immediately above the test pad, and the injected water is never evenly dispersed in the total fuel quantity. Thus, whether 100 ml or 500 ml of fuel is passed through the test pad, the Aqua-Glo should theoretically respond only to the amount of water injected. Figure 8 shows plots of the data points obtained by passing only 100 ml of fuel through the test pad but injecting equivalent amounts of water as used for the 500-ml sample runs. The abscissa in Figure 8 is listed in milligrams per liter and is equal to the quantity of water injected divided by the fuel sample size. The ordinate is the actual Aqua-Glo rating multiplied by 5 to correct for the sample size. Thus, Figures 6

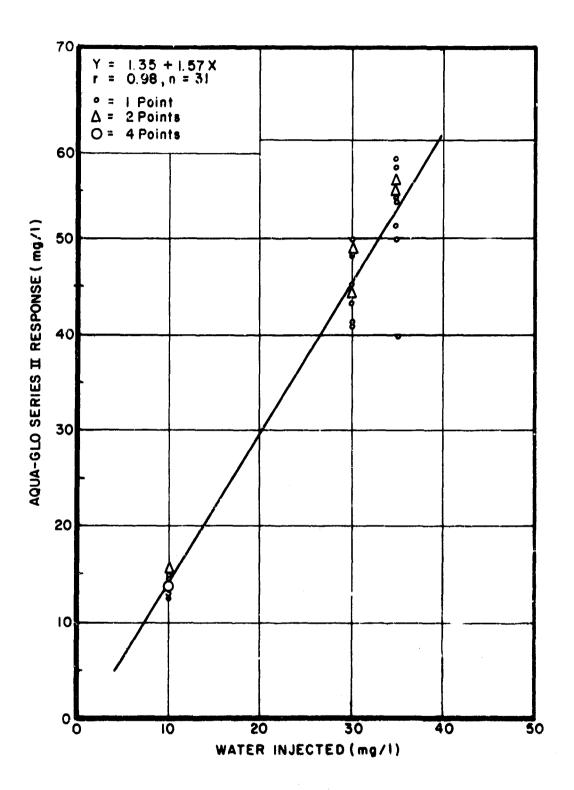


Figure 8. Aqua-Glo Response With 100-ml of JP-5 Fuel Samples Using Calibration Apparatus

and 8 can be directly compared to determine if there is any substantial difference in the Aqua-Glo response.

The regression curve obtained using the 100-ml samples was Y = -1.35 \pm 1.57X with a correlation coefficient of 0.98. This compares very well with the regression curve obtained with 500-ml fuel samples, above, Y = -0.13 \pm 1.55X. Thus, the assumption that the Aqua-Glo pads respond only to the quantity of free water and not to the quantity of fuel passed through the pads appears to be valid. This is in agreement with the manufacturer's instructions for extending the useful range of the Aqua-Glo by using smaller samples and correcting the reading for sample size.

c. Effect of Fuel Additives

A series of tests was conducted to determine the effects of jet fuel corrosion inhibitors and the antilcing additive on Aqua-Glo ratings. Three corrosion inhibitors and the fuel system antilcing additive were tested at their maximum concentrations.

- (1) Santolene C. Figure 9 shows the data obtained and the regression curve calculated -Y = -0.80 + 1.23X and r = 0.98.
- (2) AFA-1. See Figure 10. The regression curve calculated is Y = -0.18 + 1.16X and the regression coefficient (r' is 0.986.
- (3) Lubrizol 541. See Figure 1). The regression curve calculated is Y = -0.30 + 1.05X and the regression coefficient (r) = 0.98.
- (4) Fuel System 1cing Inhibitor. See Figure 12. The regression curve calculated is Y = -1.38 + 2.13X and the regression coefficient (r) is 0.987.

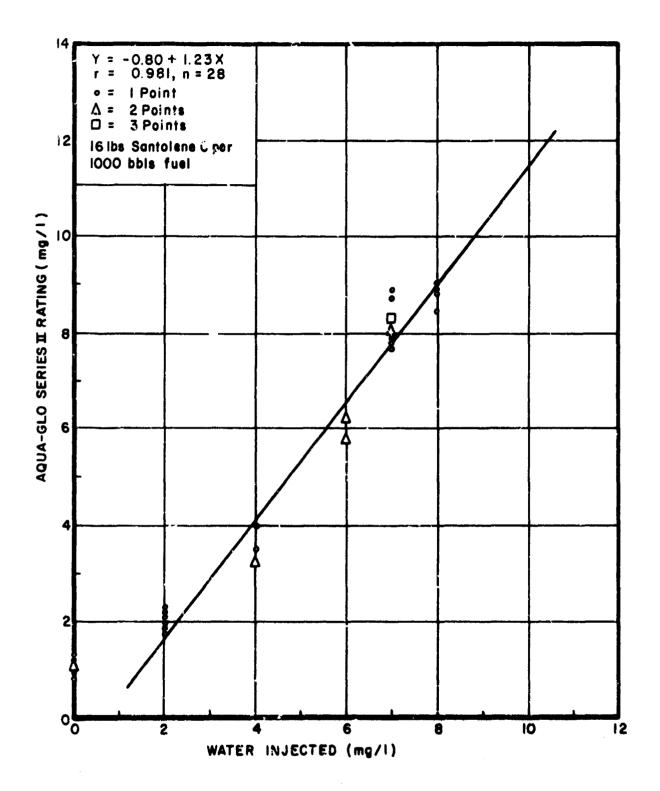


Figure 9. Aqua-Glo Response With Santolene C in JP-5 Fuel Using Calibration Apparatus

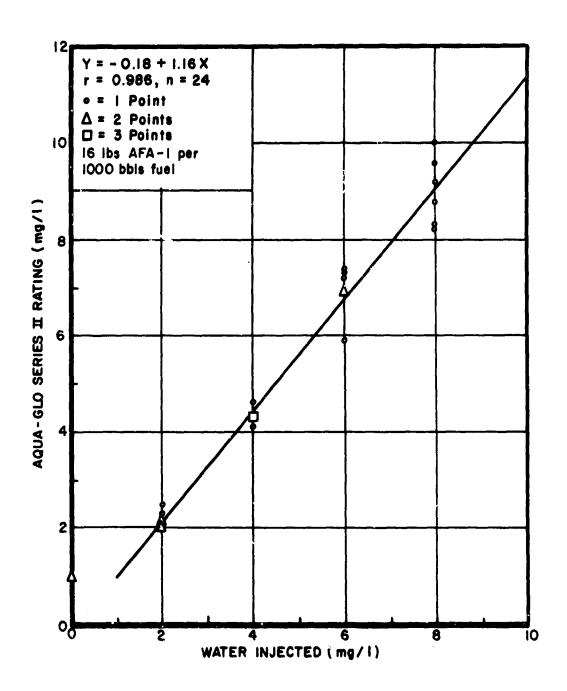


Figure 10. Aqua-Glo Response With AFA-1 in JP-5 Fuel Using Calibration Apparatus

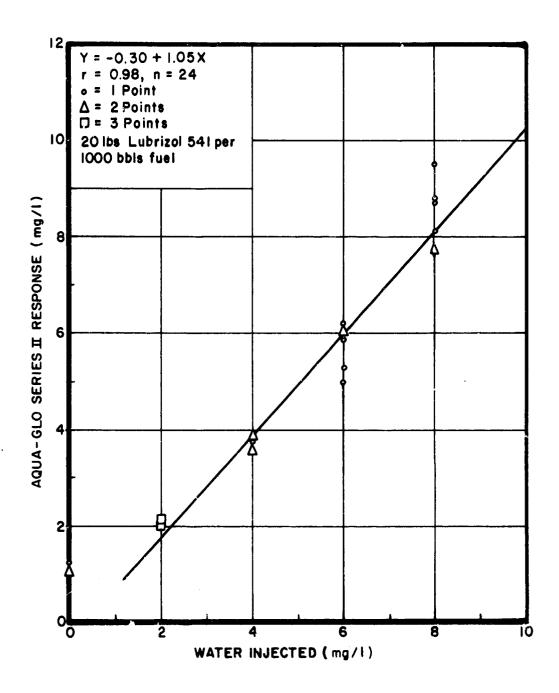


Figure 11. Aqua-Glo Response With Lubrizol 541 in JP-5 Fuel Using Calibration Apparatus

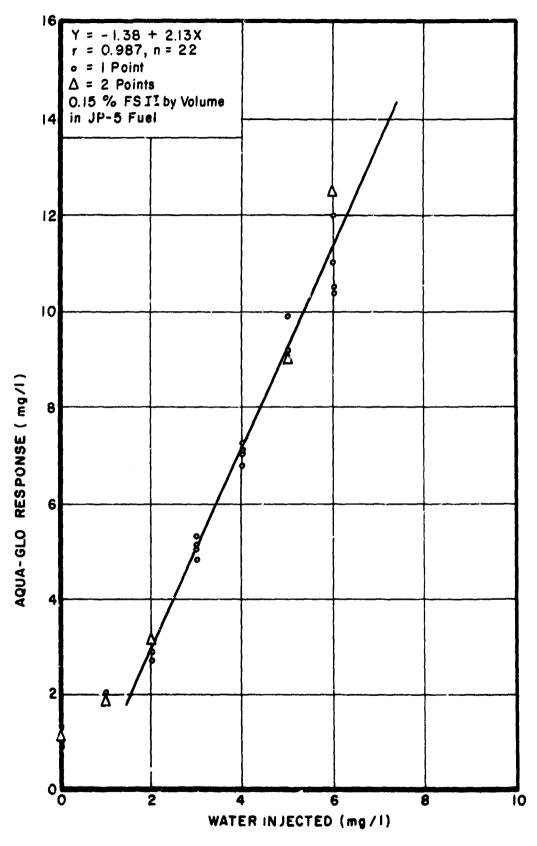


Figure 12. Aqua-Glo Response With Fuel System Icing Inhibitor in JP-5 Fuel Using Calibration Apparatus

With a 95% confidence level, it can be shown that the slopes of the regression curves for neat JP-5 and for JP-5 with additives are significantly different. That is, with only a 5% chance of being wrong, it is concluded that the corrosion inhibitors tested significantly lower the Aqua-Glo's response to free water, and the FSII significantly increases the Aqua-Glo's response to free water.

The phenomena involved in the effect of the corrosion inhibitors on the Aqua-Glo response are unknown. The FSII, however, is believed to simply dissolve in the water droplets and to increase their effective size as the FSII also tends to react with the fluorescein dye.

d. Use of 37-mm Pads

The AEL free water detector, in use by both the U.S. Navy and Air Force, uses 37-nm pads. Inasmuch as these pads are readily available, their use with the Aqua-Glo Series II was evaluated. With the Millipore 37-mm pad holder, the portion of the pad exposed to the fuel sample measures about 33-1/2 mm in diameter. The Aqua-Glo 25-mm pad holder exposes about a 19-mm diameter circle of the pad to the fuel. The ratio of these respective areas, i.e. $(33-1/2)^2/(19,^2$, is about 3.3, which is close to the correction factor of 3 recommended in the Aqua-Glo instructions.

A series of tests was conducted using the calibration apparatus with 37-mm pads and rating the pads first with the Aqua-Glo Series II (Instrument No. 1) and then visually with the AEL viewing box. Figure 13 gives the scatter diagram and the regression curve plotted for the Aqua-Glo results (the AEL results are presented in Section III-5 of this

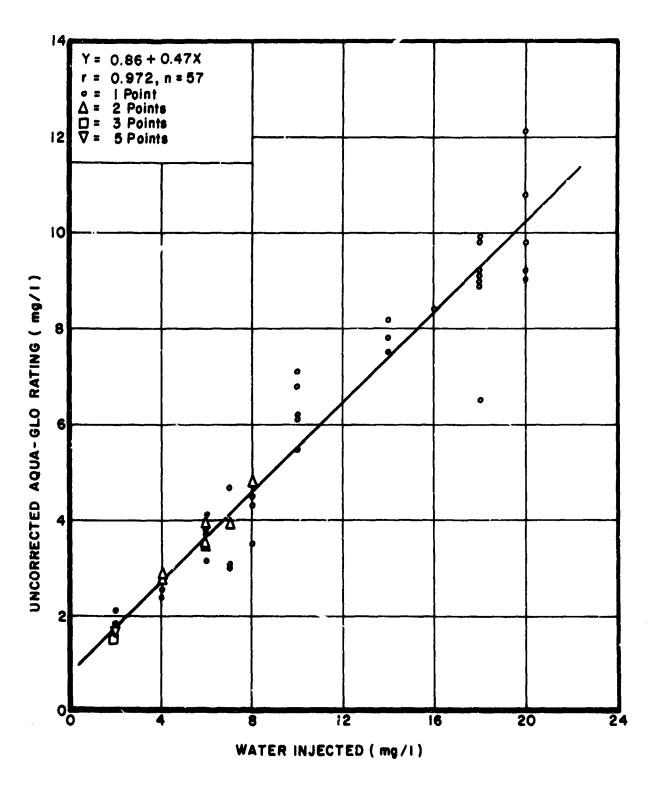


Figure 13. Aqua-Glo Response With 37-mm Pads Using Cairbration Apparatus and Neat JP-5 Fuel

report). The regression curve was calculated using all data points except for zero water injected. The regression curve equation, $Y = 0.86 \pm 0.47X$, has a correlation coefficient of 0.972, which is not as high as the regression curve correlation coefficients normally obtained with the 25-mm diameter pads. If the exposed pad area ratio of 3.3 is used, the regression curve equation becomes $Y = 2.85 \pm 1.54X$, while using the manufacturer's recommended correction factor of 3 gives the equation $Y = 2.58 \pm 1.41X$. When these regression curve equations are compared with those obtained using 25-mm pads $(Y = -0.13 \pm 1.55X)$, the regression curve slopes are fairly close, especially using the correction factor of 3.3. However, there is a difference of 2.98 $(2.85 \pm 1.41X)$ between the equation constants. The reason for this difference is unknown.

Another problem is the uneven water dispersion on the 37-mm pads. Actually, the water dispersion pattern is no worse for the 37-mm pads than for the 25-mm pads, but the Aqua-Glo rates the entire area of the 25-mm pad, and a poor dispersion pattern does not significantly affect the results. With the 37-mm pad, however, the Aqua-Glo only rates about 1/3 of the exposed area. Thus, depending upon the positioning of the 37-mm pad beneath the viewing hole, the Aqua-Glo may give substantially different ratings. This is graphically illustrated by the greater scatter seen in Figure 13 and in the lower correlation coefficient as compared to data obtained with the 25-mm pads.

2. AQUA-GLO SERIES II RESULTS USING THE LARGE TEST LOOP

Initial Aqua-Glo test results with neat JP-5 in the 600-GPM test
loops were found to vary with the fuel flow rate. This was subsequently

traced to poor dispersion of the injected water into the fuel. This was corrected by adding a small (7 GPM) centrifugal pump to the system to disperse the injected water into a small stream of fuel drawn from the main test loop. The mixture was subsequently returned to the main loop (see Figure 3). With this arrangement, the results become flow rate independent and are presented in Figure 14 for neat JP-5 and JP-5 with 2 lbs/1000 bbls AFA-1.

The Aqua-Glo response to the neat JP-5 is slightly less than that obtained in the calibration apparatus (Y = 0.04 + 1.45X for the 600-GPM loop versus Y = -0.13 + 1.55X for the calibration apparatus). The cause for this slight disagreement is unknown. Whatever the cause for the difference observed, it is not great. For example, at an injected water content of 7 mg/1, the Aqua-Glo reading using the calibration apparatus was 10.7 mg/1, while the Aqua-Glo rating with the 600-GPM test loop would be 10.2 mg/1. This difference is less than 5%.

Figure 14 also gives the data points and the regression curve for JP-5 with 2 lbs/1000 bbls of AFA-1 using the 600-GPM test loop. This regression curve is not directly comparable to data obtained with the calibration apparatus (16 lbs/1000 bbls of AFA-1 was used in the only test with AFA-1 run with the calibration apparatus). However, the regression curve slope with 2 lbs/1000 bbls of AFA-1 is less than that obtained with neat JP-5 as would be expected based on the results obtained with the calibration appratus.

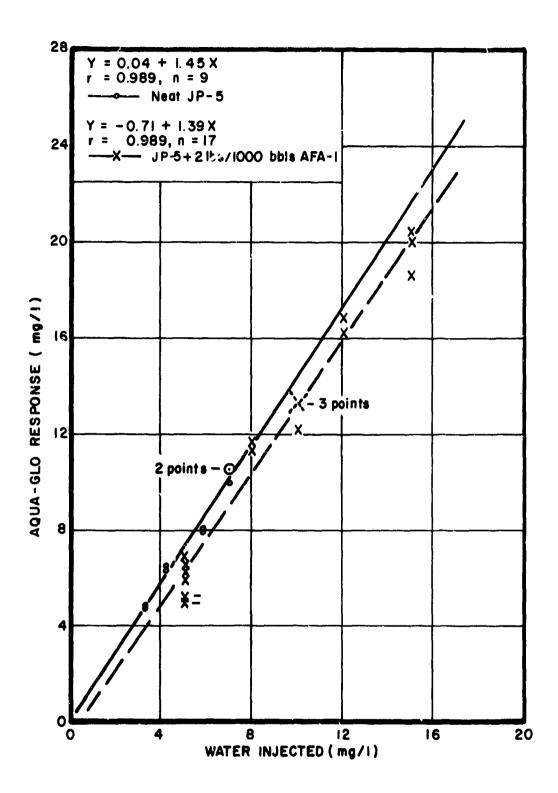


Figure 14. Aqua-Glo Response Using 600-GPM Test Loop

3. SMALL TEST 'LOOP RESULTS

a. Aqua-Gio Series II Results

Three test runs were conducted using the 38-GPM test loop; one with clay-treated JP-5, one with JP-5 containing 4 lbs/1000 bbls of Santolene C, and one containing 0.15% FSII. The test results are presented in Figures 15 and 16. The Aqua-Glo data with clay-treated JP-5 resulted in a regression curve of Y = -0.13 + 1.51X which agrees very well with that obtained with neat JP-5 with the calibration apparatus (Y = -0.13 +1.55%). With 0.15 FSII in the JP-5, the small test loop Aqua-Glo results gave a regression curve of Y = -1.69 + 2.02X. This also compares favorably with that obtained with the static calibration apparatus with 0.15% FSII in JP-5 (Y = -1.38 + 2.13X). With 4 lbs/1000 bbls Santolene C, the small loop Aqua-Gio results give a regression curve of Y = -0.91 + 1.1X. This does not compare well with that obtained with the static calibration apparatus with 16 lbs/1000 bbls Santolene C (Y = -0.80 + 1.23X), since the results with 4 lbs/1000 bbis of Santolene C would be expected to give a higher Aqua-Glo response than would the 16 lbs/1000 bbls results.

One interesting result with Santolene C present in the fuel is noted in Figure 16. Note that the Aqua-Glo response increased with time (the chronological order of the data points taken is indicated by the arrows). The cause for this change in response is uncertain; it could have been caused by uneven dispersion of the Santolene C in the fuel (which had only been mixed for 15 minutes after addition of the Santolene C), or the Santolene C may have been partly removed from the system-reither by adsorption on pipe and tank walls or by the clean-up filter-separator in the test loop.

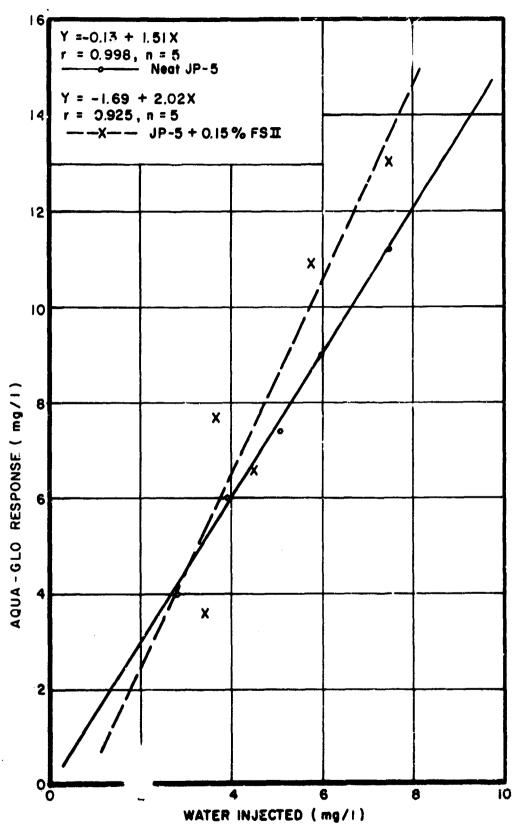


Figure 15. Aqua-Glo Response to Neat JP-5 and JP-5 Plus 0.15% Fuel System Icing Inhibitor Using 38-GPM Test Loop

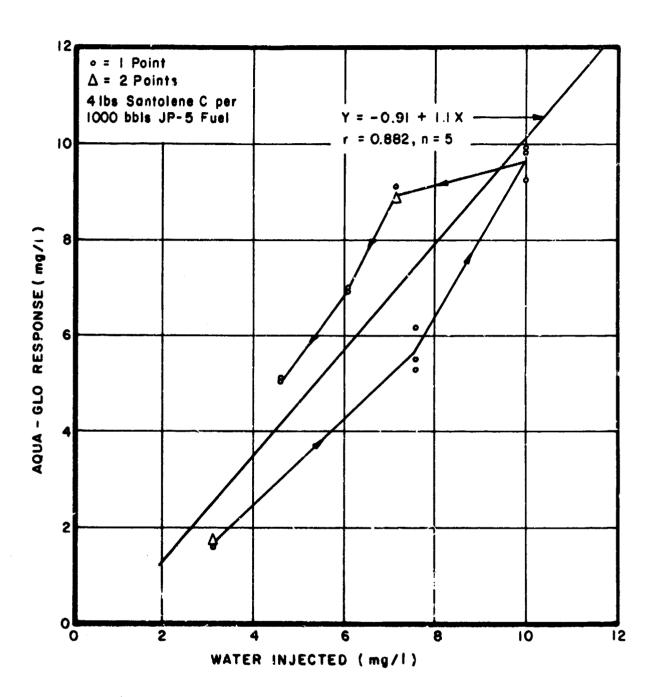


Figure 16. Aqua-Glo Response With Santolene C in JP-5 Fuel Using 38-GPM Test Loop

b. Keene Turbidimeter and Bowser Totamitor Results

During the small loop calibration runs, both a Bowser Totamitor and a Keene Turbidimeter were also used. Neither the presence of Santolene C nor FSII significantly affected the response of the Totamitor, but a slight reduction in the Turbidimeter response was noted when the fuel contained the FSII. See Figures 17 and 18. As both the Turbidimeter and the Totamitor detect free water by light scattered by the water droplets in the fuel, it appears that Santolene C and FSII have little if any effect on the degree to which the water is dispersed in the fuel. Since the fuel additives do affect the Aqua-Glo response, however, this may be interpreted as being caused by a chemical phenomenon.

FSII (99.6% ethylene glycol monomethyl ether and 0.4% glycerol) was found to react with the fluorescein dye on the Aqua-Glo pads much the same as does water. Since the FSII has a very high affinity for water, it is postulated that the water droplets dispersed in the fuel absorb significant quantities of the FSII and that both the water and the water-absorbed FSII then mach with the Aqua-Glo pad. How such an absorption of FSII into the water droplets would affect the Turbidimeter and Totamitor response is uncertain, however.

4. POSSIBLE ERRORS IN USE OF AQUA-GLO SERIES II

a. Comparison of Two Aqua-Glo Series II Instruments

Two Aqua-Glo instruments were available for use in this program. The majority of the results were obtained using instrument No. 1. The two instruments were compared by drying used 25-mm pads in a dessicator and then rating using both instruments. (By using dry pads, the change

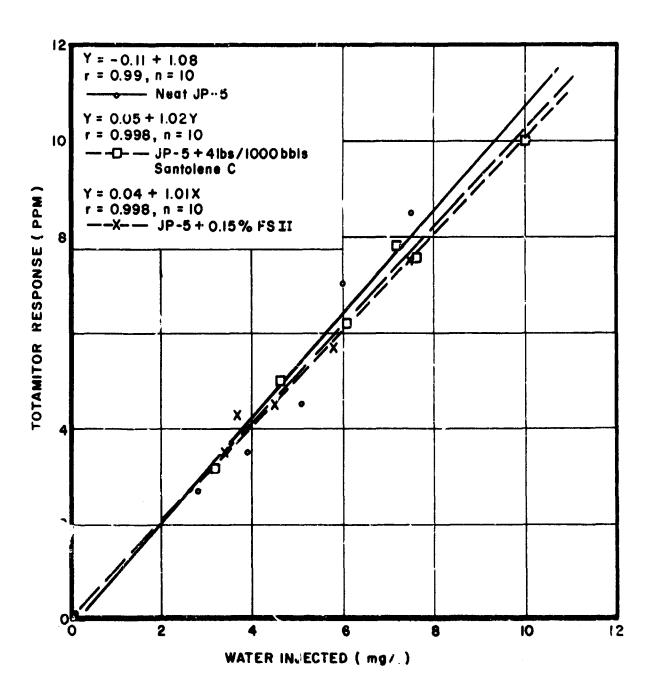


Figure 17. Totamitor Response Using 38-GPM Test Loop

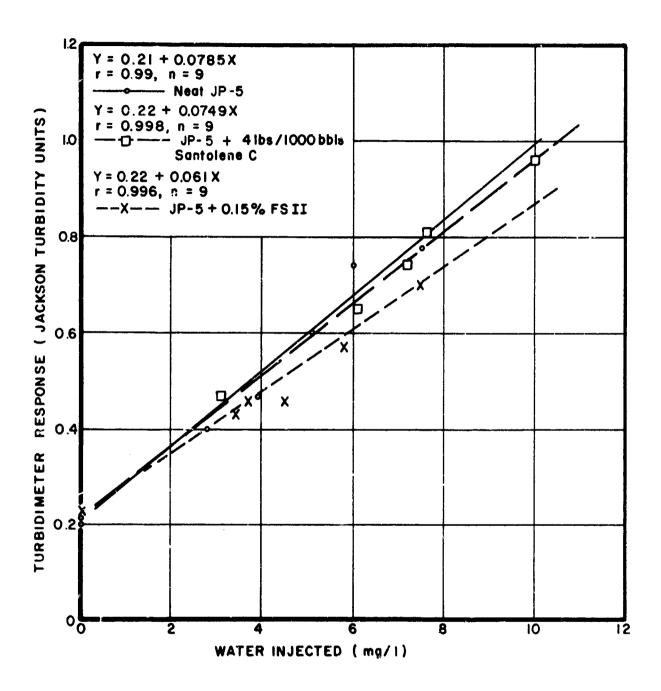


Figure 18. Turbidimeter Response Using 38-GPM Test Loop

in the pads with time was avoided). Figure 19 shows plots of the data points obtained with the dry pads for the two instruments. The correlation curve has the equation Y = 0.0766 + 0.886X where Y is Instrument No. 2 rating and X is Instrument No. 1 rating. The correlation coefficient for this curve is 0.998-excellent correlation.

Figure 19 indicates that substantial differences in the accuracy of two or more Aqua-Glo Series II instruments may be found. Thus, for maximum accuracy each instrument must be individually calibrated. The very slight scatter seen in the data points in Figure 19 indicates, however, that good reproducibility with the Aqua-Glo Series II is possible.

b. Comparison of Wet Versus Dry Pad Ratings

The Aqua-Glo Series II instructions (Reference 4) state that if the Aqua-Glo pads are allowed to dry in a dessicator and then rated, the rating must be corrected by dividing the reading by 1.2 for water contents of 1 to 12 PPM and by 1.3 PPM for water contents greater than 12 PPM. Figure 20 shows plots of the data points and the correlation curve for the Aqua-Glo ratings of wet pads and the same pads after drying in a dessicator. The correlation curve equation is Y = -0.283 + 1.52X and the correlation coefficient is 0.9815. The slope of 1.52 indicates that the dry pads read about 52% higher than do the wet pads. The dried pad rating can be converted to the equivalent wet pad rating by multiplying the dried pad rating by a correction factor of 1/1.52 or about 0.66. This compares to the correction factor of $\frac{1}{1.2} = 0.83$ recommended by the Aqua-Glo Series II instructions (Reference 4).

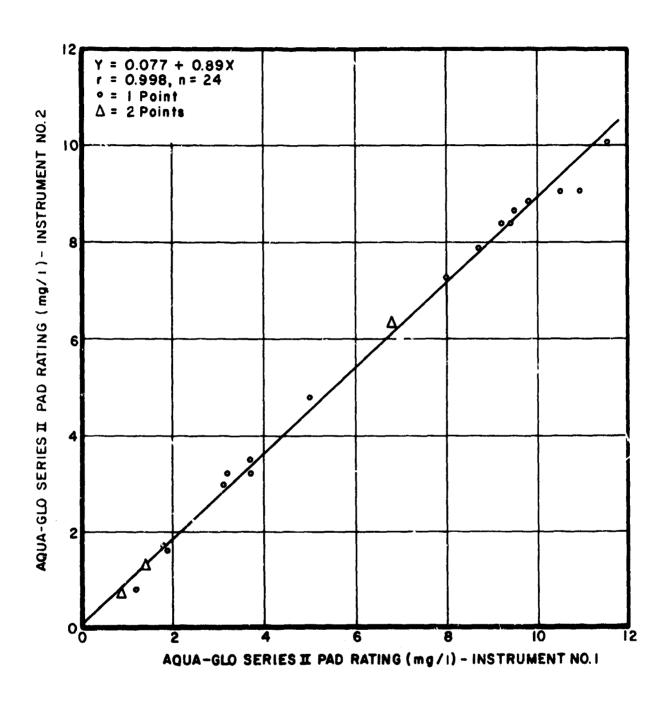


Figure 19. Comparison of Two Aqua-Glo Series II Instruments

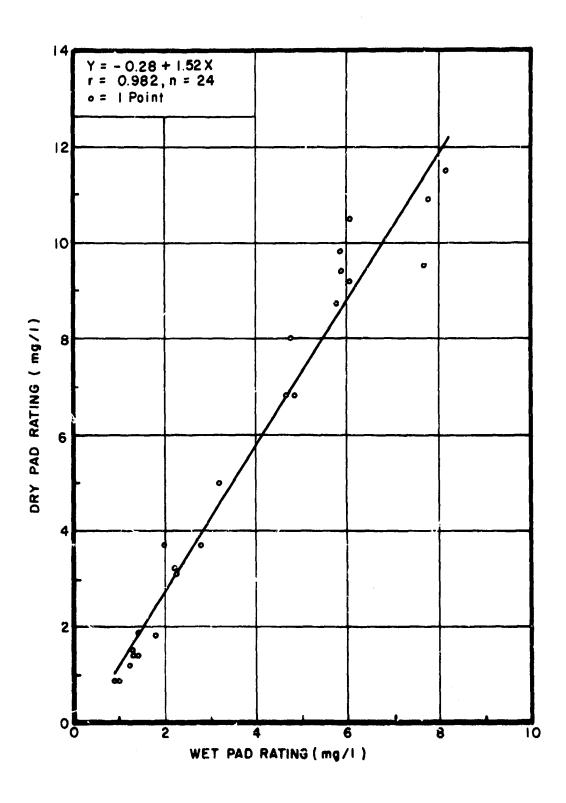


Figure 20. Comparison of Wet Pad With Dry Pad Aqua-Glo Ratings

Note that when neat JP-5 fuel is used and the pads are dried, the total correction factor would be the dried-pad-to-wet-pad correction factor multiplied by the wet pad calibration factor of 0.64 (see Figure 7 and the regression equation of X = 0.14 + 0.64Y). Thus, for Instrument No. 1, the total correction factor for dried pads and neat JP-5 would be $0.66 \times 0.64 = 0.42$. That is, with neat JP-5 and drying the Aqua-Glo pads in a dessicator prior to rating, the Aqua-Glo Series II reading must be multiplied by 0.42 to obtain the actual free water content of the fuel in milligrams per liter.

c. Aqua-Glo Pad Dye Content Effects

Used Aqua-Glo pads, having been exposed to JP-5 fuel with water contents ranging from 1 to 8 mg/1, were subsequently measured for uranine dye content using the procedure described in the Appendix. A regression curve incorporating two sets of data was calculated, $Y_{ave} = -0.004 + 1.36X$, where X is the actual free water content and Yave is the average Aqua-Glo response. The difference between the actual Aqua-Glo response for each pad (Yact) and the calculated Aqua-Glo response based on the regression equation (Y_{ave}) was designated as ΔY ($\Delta Y = Y_{act} - Y_{ave}$). The ratio of Δ Y/Y $_{
m act}$ was then plotted against the actual pad dye content and is presented in Figure 21. In addition, regression curve equations were calculated for both sets of data and these regression curve equations and their correlation coefficients are also presented in Figure 21. Note that the slope of one of the regression curves is slightly positive, the slope of the other slightly negative, and that the correlation coefficients for both curves are very low. This is interpreted to mean that there is no significant relationship between the Aqua-Glo response and the pad dye content for the range of pad dye contents covered (0.047 to 0.073 mq/cm^2).

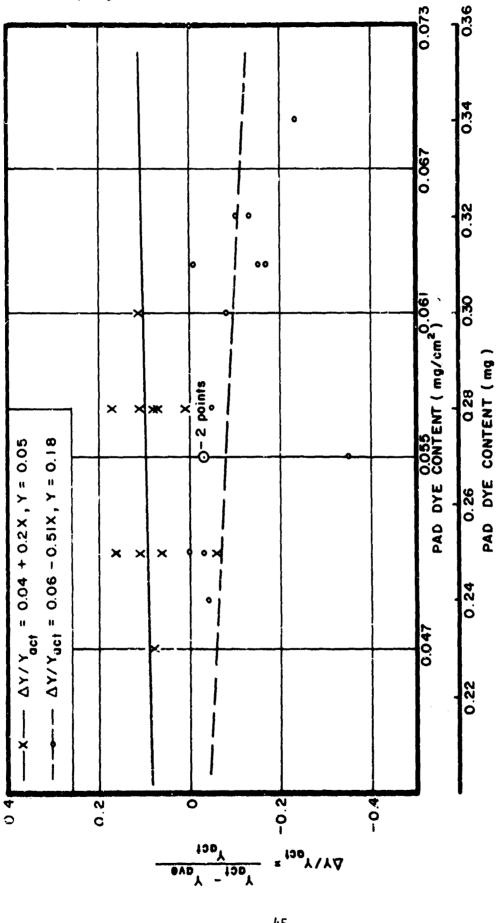


Figure 21. Aqua-Glo Rating Versus Pad Dye Content

The military specification for AEL free water detector pads requires 0.80 to 0.85 mg of uranine dye per 47-mm pad, equivalent to 0.046 to 0.049 mg/cm². Table I presents the measured pad dye content for various AEL and Aqua-Glo pads. Note that only one pad was found to have a dye content below 0.046 mg/cm² and it was 0.045 mg/cm². Many pads, including those in Figure 21, have pad dye contents well above 0.049 mg/cm² as specified by MIL-D-81248. It appears that MIL-D-81248 is unduly rigid as only a lower limit on the pad dye content may be needed, and 0.046 mg/cm² appears to be an acceptable lower limit.

d. Time Interval Between Sampling and Reading

As noted in the procedures for using the calibration apparatus (Section II-1), the time interval between start of sampling and the reading of the Aqua-Glo pad affects the rating value. Throughout the test program, a time of 3 to 3-1/4 minutes was maintained between start of sampling and pad rating. This time interval gave adequate time to sample the fuel; to remove the pad from the holder, blot four times, and insert into the Aqua-Glo; and to allow 60 to 70 seconds for the photocell detector assembly to stabilize before reading. By extending the time interval between sampling and pad reading to 5 to 10 minutes, the Aqua-Glo reading was found to decrease by 1 to 2 mg/l. However, vien the pads are dried in a dessicator, the reading of the dry pad would be considerably higher. Thus, at some time interval greater than 10 minutes, the readings must start to increase.

One critical aspect of the Aqua-Glo and AEL pads, however, is opposed them from moisture. As received, the pads are packaged in vapor-

TABLE I
PAD DYE CONTENT

PAD DYE CON	ITENT	
Pad Size and Identification	Total Milligrams Uranine on Pad	Milligrams Uranine/cm ²
47-mm diameter — Drivire, Inc. MILD-81248	0.89	0.051
Mir Part No.375	1.08	0.062
NI56-47179	1.20 1.17	0.069
Date of manufacture 12/67	1.05	0.067
37-mm diameter - Driaire, Inc.	• • •	
MIL-D-81248	0.61	0.057
Mfr Part No. 376	0.75 0.69	0.070
6630-105-4386	0.55	0.064
Date of manufacture 6/69	0.63	0.051
	0.00	0.059
25-mm diameter - Gammon Technica!	0.38	0.077
Products, Inc.	0.43	0.088
Date of manufacture 8/66		1
25-mm diameter — Gammon Technical	0.24	0.049
Products, Inc.	0.22	0.045
Date of manufacture 10/67	0.30	0.061
	0.31	0.063
	0.33	0.067
25-mm diameter - Gammon Technical	0.27	0.055
Products, Inc.	0.31	0.063
Date of manufacture 4/69	0.34	0.069
	ე.35	0.071
	0.27	0.055
25-mm dianieter - Gammon Technical	0.28	0.057
Products, Inc.	0.28	0.057
Date of manufacture 4/69	0.25	0.051
USED PADS	0.28	0.057
	0.25	0.051
	0.23	0.047
	0.23	0.047
İ	0.28	0.057
	0.25	0.051
	0.25	0.051
1	0.30 0.25	0.061
Į.	0.28	0.051
1	0.28	0.057
j	0.30	0.057
Í	0.28	0.061
		0.057

TABLE I (Contd)

Pad Size and Identification	Total Milligrams Uranine on Pad	Milligrams Uranine /cm²
25-mm diameter — Gammon Technical Products, Inc.	0.27 0:34	0.055 0.069
Date of manufacture 4/69	0.31	0.063
Used Pads	0.32 0.32	0.065 0.065
	0.31 0.31	0.063 0.063
	0.28	0.057
1	0.24 0.27	0.049 0.055
	0.27	0.055
	0.25 0.25	0.051 0.051
	0.36 0.30	0.073 0.06 I

Note: Military Specification for AEL pads require 0.80 to 0.85 mg uranine dye per 47-mm pad, equivalent to 0.046 to 0.049 mg/cm².

proof envelopes. When given reasonable storage protection, the pads remain usable for one or more years. However, once the pad is removed from its protective envelope, it may absorb moisture, depending upon the relative humidity. Also, any source of water droplets must be avoided. For example, sneezing or coughing in the vicinity of the exposed pad may result in high readings.

Before use, each pad should be visually examined. A good, fresh pad is yellow-orange, and the color will slowly lighten to yellow when exposed to the atmosphere. Any evidence of color spotting may indicate that the pad has been exposed to water droplets. If there is any doubt as to the pad quality, it should either be discarded or checked in the

Aqua-Glo or an AEL viewing box before use. Checks of fresh Aqua-Glo pads normally gave an Aqua-Glo rating of less than 1 to about 1.3. AEL and Aqua-Glo pads should have no visible fluorescence when viewed in the AEL viewing box, i.e., they should have an even, dark purple, or violet color when illuminated by the ultraviolet light.

e. Other Possible Causes of Error

As some jet fuels have been found to fluoresce under untraviolet light, it was speculated that the natural fluorescence of the fuel would be sufficient to cause an error with the Aqua-Glo or the AEL. Therefore, four JP-4 fuel samples were selected with two samples exhibiting considerable fluorescence, one moderate fluorescence, and one very little fluorescence. For each of the four fuels, four or five Aqua-Glo pads had 500 ml of the fuel passed through them to determine if there was any observable increase in the meter reading. The mean for each of the four fuels was between 0.92 and 0.98 with the maximum and minimum mean obtained with the two fuels having the highest degree of fluorescence. Thus, jet fuel fluorescence would not be likely to affect the accuracy of the Aqua-Glo or AEL free water detectors. The Aqua-Glo instructions state, however, that fuel fluorescence may be a problem with oils and heavy fuels (Reference 4).

The Aqua-Glo instructions state that the optical surfaces within the Aqua-Glo must be periodically cleaned (Reference 4). Although no measurable changes in the calibration of the Aqua-Glo were noted before and after considerable dust was removed from the bottom glass filter, care should be taken to keep the instrument's optical surfaces clean.

Another possible cause of error is the leakage of light into the instrument. The bottom flap covering both the permanent standard and the test pad is a potential light leakage area. The slot for the nulling control/readout lever is another possible light leakage area. A simple check for light leaks can be accomplished by zeroing the instrument with the calibration standard in place and varying the intensity of the ambient light. Any change in the instrument calibration suggests a light leak. Subsequent to the test program reported herein, light leaks were found in both Instruments 1 and 2. This indicates that the Aqua-Glo Series II may be capable of better accuracy and repeatability than was obtained.

5. AEL FREE WATER DETECTOR RESULTS

Varying amounts of free water were injected into neat JP-5 immediately upstream of 37-mm AEL pads using the calibration apparatus. The results of this series of tests are presented in Figure 22 which consist of a scatter diagram of all points and the regression curve plotted using all data points. The regression curve Y = 2.56 + 1.86X has a correlation coefficient of 0.93 and the true slope of the regression curve for the entire population lies between 1.75 and 1.97 with a confidence level of 95% assuming a normal sampling distribution.

The considerable scatter is believed due primarily to the use of the eye to compare the pad fluorescence with the AEL standards. With varying water dispersions and distribution patterns on the AEL pads, the task of visually comparing the total pad brightness to the standards is subjective and difficult.

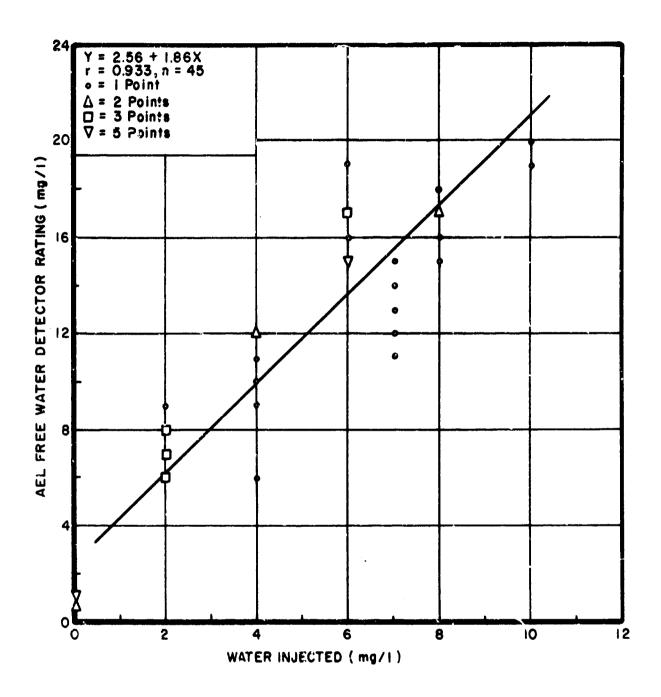


Figure 22. Calibration Check of AEL Free Water Detector Using 500-ml Samples and Calibration Apparatus

Southwest Research Institute (Reference 2) conducted a similar calibration check with the AEL free water detector. Using a batch-blending system, it obtained a regression curve of Y = -0.7 + 1.86X. This compares very well with the results obtained above with a difference of about 3 mg/l over the range of 0 to 20 mg/l. Based on this excellent agreement, we concur in the Southwest Research Institute's conclusion that a 275-ml sample (i.e., 500 ml/ $\frac{1}{2}$.86) should be used in lieu of the sandard 500-ml sample for maximum accuracy.

SECTION IV

SUMMARY AND CONCLUSIONS

1. SUMMARY

The accuracy of the Aqua-Glo Series II free water detector was checked using neat JP-5, JP-5 with three different corrosion inhibitors, and JP-5 with 0.15% fuel system icing inhibitor (FSII). Three methods for measuring the accuracy of the Aqua-Glo water used: a small calibration apparatus in which a known amount of free water was injected immediately above the Aqua-Glo pad into a flowing fuel stream, a 38-GPM test loop, and a 600-GPM test loop wherein known amounts of free water were injected upstream of the sample port.

Since there is no accepted Standard Calibration Method for use with free ater detectors such as the Aqua-Glo Series II, the results of the three calibration methods used are assumed to be approximately correct inasmuch as they agree fairly well. Listed in Table II and plotted in Figure 23 are the linear regression curves (Y = Aqua-Glo reading in mg/l and X = amount of water injected into the fuel in mg/l) calculated using the data points obtained during the tests. Following each regression curve is the correlation coefficient obtained for that regression curve.

In general, the regression curves agree quite closely for each fuel/fuel additive blend. When the results of the three calibration methods are compared, it is seen that the regression curves for neat CP-5 agree quite well with a range of values of 6.4 to 7.6 mg/l for an actual injected water quantity of 5 mg/l-this amounts to about 19% difference.

TABLE II

SUMMARY OF AQUA-GLO REGRESSION CURVES

Calibration Method Used	Fuel Composition	Regression Curve	Correlation Coefficient
a. Calibration Apparatus	Neat JP-5 (500-ml sample)	Y = -0.13 + 1.55X	0.993
:	2	Y = -0.30 + 1.34X	966.0
ij	Neat JP-5 (100-ml sample)	Y = -1.35 + 1.57 X	96.0
d. 600-GPM Test Loop	Neat JP-5	Y = 0.04 + 1.45X	0.989
e. 38-GPM Test Loop	Neat JP-5	Y = -0.13 + 1.51 X	0.998
f. Celibration Apparatus	JP-5 + 161bs / 1000 bbis Santolene C	Y = - 0.80 + 1.23X	- E6 O
g. 38-GPM Test Loop	JP-5 + 4 lbs/1000 bbis Santolene C	Y = -0.91 + 1.1X	0.882
h. Calibration Apparatus	JP-5+ I6 lbs/1000 bbls AFA-1	Y = - 0.18 + 1.16X	0.986
i. 600-GPM Test Loop	JP-5 + 2 lbs/1000 bbis AFA - I	Y = -0.71 + 1.39X	0.989
j. Calibration Apparatus	JP-5 + 20 lbs/1000 bbls Lubrizol	Y = -0.30 + 1.05 X	0.930
=	JP-5 + 0.15% FSII	Y = -1.38 + 2.13X	7.86.0
I. 38-GPM Test Loop	JP-3 + 0 15% FSII	Y = - 1.69 + 2.02X	0.925

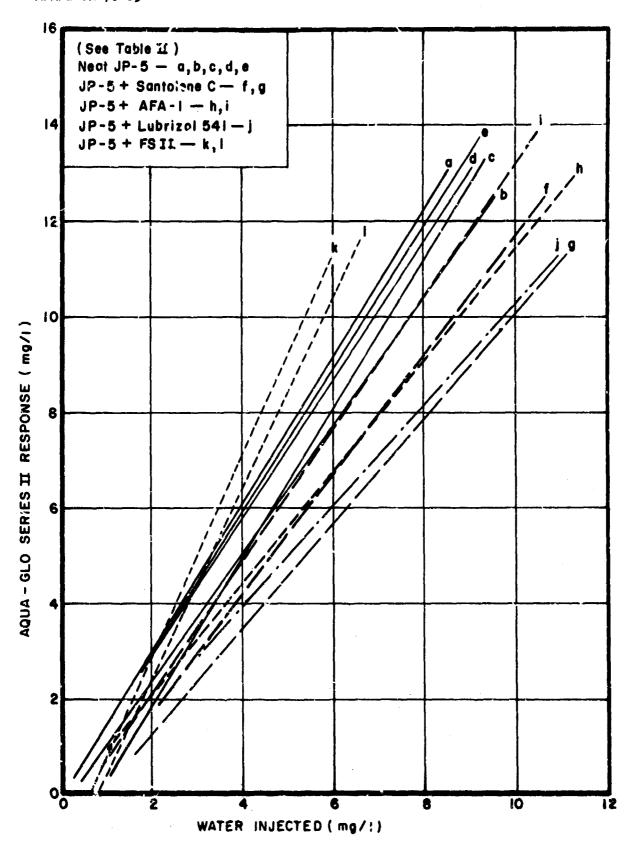


Figure 23. Summery of Aqua-Glo Series II Regression Curves

The only other set of regression equations directly comparable is for JP-5 + 0.15% FSII; here the difference is about 11% for 5 mg/l water injected.

The reasons for these differences are unknown, but may include one or more of the following:

- 1. Errors in measurement, especially in the accuracy of the amount of water injected when the rotometers are used on the two test loops.
- 2. Differences in fuel composition--neat JP-5 may vary considerably from one batch to another and even within the same batch with time. It has been shown that fuel additives significantly affect the Aqua-Glo response, and the neat JP-5 may have picked up trace amounts of fuel additives and other contaminants, that could affect the Aqua-Glo response.
- 3. Change of additive concentration in the fuel with time. Corrosion inhibitor additives are designed to plate out on fuel system surfaces to protect against corrosion. Thus, a loss of the corrosion inhibitors with time could occur by adsorption of the additive onto surfaces.
- 4. Differences in the calibration methods. The calibration apparatus used does not allow the injected water to reside in the fuel for more than a fraction of a second while the test loops may allow the water to reside in the fuel for up to 3 or 4 seconds, depending upon flow rate. Changes at the fuel/water interface are believed to require substantial time for equilibrium conditions to occur, and none of the three calibration methods used may closely approximate field conditions in this respect.
- 5. Light leakage in the Aqua-Glo Series II. Subsequent to the test program light leaks were found in both instruments.

2. CONCLUSIONS

- a. The Aqua-Glo Series II free water detector has been found to be quite sensitive to free water in JP-5 fuel and has demonstrated excellent repeatability. Similar performance with JP-4 and other hydrocarbon fuels would be expected. However, for maximum accuracy, the Aqua-Glo Series II must be calibrated with each fuel/fuel-additive blend. The two Aqua-Glo Series II detectors tested read high by 40% to 50% using neat JP-5.
- b. The useful, accurate range of the Aqua-Glo Series II free water detector is from about 2 to 8 mg/l using 500-ml fuel samples. Below about 2 mg/l the scale becomes nonlinear and above about 8 mg/l the Aqua-Glo reads off scale. However, when sample size is changed, the range can be increased. For example, with 100-ml fuel samples, the useful range would be between 10 and 40 mg/l. Although not attempted, it should be possible to use larger fuel samples to extend the range below 2 mg/l; for example, a 1000 mg/l fuel sample should give a useful, accurate range of 1 to 4 mg/l.
- c. The Aqua-Glo Series II, when used with 37-mm pads, is not as accurate as when used with 25-mm pads. However, the Aqua-Glo could be modified to use 37-mm pads with equivalent accuracy.
- d. The Aqua-Glo Series II is clearly superior to the AEL free water detector in terms of accuracy, repeatability, and portability. However, it is slightly slower in use as it takes about 50 to 80 seconds for the photocell detector assembly to stabilize each time it is used.
- e. The accuracy and repeatability of the Aqua-Glo Series II (and probably any other type of detector that uses the fluorescein treated pads)

is improved by using a predetermined time interval between start of sampling and pad rating.

- f. The calibration of the Aqua-Glo Series II should be made using the same operating procedure as will be used during its use. The rating of pads after they have been dried in a desiccator is permissible if the instrument has been calibrated using the dried pads. Otherwise, correction factors must be used.
- g. The calibration apparatus, described in Section II-i appears to be a satisfactory means for calibrating the Aqua-Glo Series II and the AEL free water detectors. Its use with free water detectors that rely on a homogeneous fuel/water emulsion is not recommended.
- h. For maximum accuracy of the Aqua-Glo or AEL free water detectors, the free water in the fuel must be well dispersed in the fuel. Large water droplets which contact the pad will tend to dissolve the fluorescein dye and allow it to penetrate deep into and through the pad giving low readings. The fine or very small water droplets, however, do not penetrate as deeply and cause a greater fluorescence on the pad surface.
- i. Possible light leaks in the Aqua-Glo Series II should be checked periodically. This is especially important for Aqua-Glo Series I instruments converted to the Series II configuration.

SECTION V

RECOMMENDATIONS

- i. The Aqua-Glo Series II free water detector is recommended as an alternate to the AEL free water detector for field use. For field use, its primary advantages over the AEL free water detector are increased portability, battery-powered operation, and a more accurate read-out technique. However, unless calibrated for use with the particular fuel/fuel additive blend in question, the AEL and the Aqua-Glo Series II may read 50% to 100% high, i.e., if the Aqua-Glo reading is 10 mg/l, the actual water content may be only 5 to 7 mg/l.
- 2. The Aqua-Glo Series II free water detector is recommended for use a laboratory instrument if carefully calibrated using the test fuel/fuel additive blend and if a standard time interval is used between sampling and reading. The Aqua-Glo range can be extended by varying the fuel sample size. The useful range of the Aqua-Glo Series II was found to be about 2 to 40 mg/l using sample sizes between 500 and 100 ml.
- 3. The Aqua-Glo Series II can be used with 37-mm pads, but a substantial decrease in accuracy will result. However, if the Aqua-Glo Series II is to be used exclusively with 37-mm pads, it would be worthwhile to modify the instrument so that the entire fuel-exposed surface of the pad is viewed by the photocell in the readout assembly.

 Recalibration of the Aqua-Glo would be necessary after such a modification.
- 4. When using the Aqua-Glo Series II, the operator should occasionally visually examine the water dispersal pattern on the test pads.

If only a few large fluorescence spots are seen rather than many small spots or a general glow over the entire pad, the water is not well dispersed and subsequent Aqua-Glo Series II readings may be low. With a poor water dispersion, erroneous readings may also be obtained with the AEL free water detector and with continuous fuel monitors which rely on light scattering detection techniques such as the Keene Turbidimeter.

5. The validity of the calibration techniques used in this report for calibrating the Aqua-Glo Series II should be checked by other agencies. If found to be sufficiently accurate, the calibration apparatus described in Section !I-l is recommended as a simple calibration method for use with the Aqua-Glo Series II and the AEL free water detectors.

APPENDIX

PROCEDURE FOR DETERMINING THE TOTAL DYE CONTENT FOR AQUA-GLO AND AEL PADS

The total dye content of each pad was found by spectrophotometric comparison of the water extracts from each pad with standard solutions of sodium fluorescein (uranine). Solutions of uranine in water were prepared in concentrations of 0.5, 1.0, 1.5, and 2.0 mg/l. A maximum absorbance plateau for the uranine was found at 455 m μ on a wavelength scan, so this was chosen as the wavelength for making all absorbance measurements. Absorbance measurements were made at the 455 mu setting on the Bausch and Lomb Spectron: 20 using the standard 1.2 cm cells for this instrument. The standard curve is included as Figure 24. The uranine content of each pad was determined by washing the pad with five 40-50 ml washes of water which were quantitatively transferred to a volumetric flash and diluted to volume with water. In order to obtain absorbance readings of mid-range intensity, different dilutions were used for the 25. 37, and 47 mm pads (250 ml, 500 ml, and 1000 ml, respectively). A portion of the solution was filtered through a dry Whatman No. 12 paper and its absorbance at 455 m \(\mu \) measured in the 1.2cm cells of the Spectronic -20. The uranine concentration was obtained from the calibration curve and the content of each pad was calculated as follows:

Mg uranine in pad = CV

where

C = concentration of uranine in milligrams per liter (from curve)

V = total volume of solution in liters

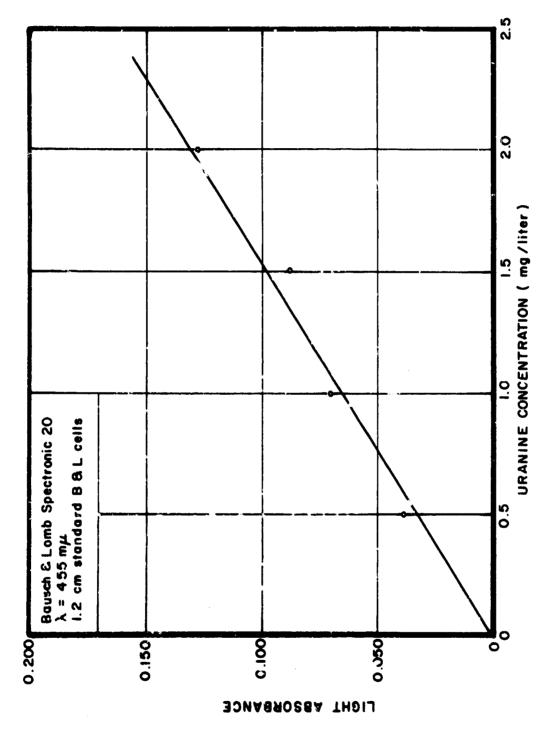


Figure 24. Uranine Dye Concentration Versus Light Absorbance

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The Aqua-Glo Series II free water detector, for use in measuring the quantity of free (undissolved) water in hydrocarbon fuels, was evaluated. Two dynamic test loops and a batch-blending calibration apparatus were used to check the accuracy and repeaturility of the Aqua-Glo Series II. The Aqua-Glo's response to free water in fuel was found to be affected by the presence of fuel additives in the fuel and by the degree to which the water was dispersed in the fuel. Corrosion inhibitors such as Santolene C, AFA-1, and Lubrizol 541 were found to decrease the Aqua-Glo's response to free water while the fuel system icing inhibitor was found to increase the Aqua-Glo's response to free water. A poor dispersion of the free water in fuel, i.e., one that has a few large drops versus a good dispersion, where there are many small drops, resulted in decreased repeatability and a lowered Aqua-Glo response.

In general, the Aqua-Glo Series II free water detector was found to be suitable for field use in its present configuration over the range of 2 to 40 milligrams per liter of water in the fuel. For laboratory use, the accuracy can be maintained within ±10% if the Aqua-Glo is first calibrated for use with each particular fuel/fuel-additive blend. The Aqua-Glo Series II is superior to the AEL free water detector in terms of accuracy, repeatability, and portability.

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