

HYDROSCOUT[®]

For the Determination of Water Content

User's Manual

Version E34

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WARNING!

READ ALL CAUTIONS BEFORE USING THIS TEST!

ONCE THE TEST IS INITIATED, CONTENTS OF THE TUBES ARE UNDER PRESSURE. SAFETY GLASSES MUST BE WORN AT ALL TIMES WHILE PERFORMING TESTS. DO NOT REMOVE THE WHITE CAP AFTER THE TEST HAS BEEN PERFORMED.

DO NOT USE THIS TEST ON CONCENTRATED ACIDS. THE EXCESSIVE HEAT GENERATED MAY LEAD TO TUBE FAILURE.

DO NOT USE 5 ML SAMPLES OF PHOSPHATE ESTER-BASED HYDRAULIC FLUIDS, GASOLINE OR SOLVENTS SUCH AS ACETONE. THESE SAMPLES WEAKEN THE TUBES WHICH MAY RESULT IN TUBE FAILURE.

THE TEST CONTAINS FLAMMABLE SOLVENTS AND PRODUCES HYDROGEN GAS; KEEP AWAY FROM OPEN FLAME.

MAKE SURE THE BLACK SEPTUM IS FULLY INSERTED INTO THE NECK OF THE TEST TUBE AND THE WHITE CAP IS SCREWED ON TIGHTLY.

SHAKE THE TUBE WHILE POINTING IT AWAY FROM YOURSELF AND BYSTANDERS.

WHEN CRUSHING THE GLASS AMPULE, PRESS FIRMLY IN THE CENTER OF THE GLASS AMPULE. NEVER ATTEMPT TO RECRUSH BROKEN GLASS AS IT CAN PENETRATE THE PLASTIC TUBE AND CUT FINGERS.

THE GRAY AMPULE IN THE TUBES CONTAINS CALCIUM HYDRIDE WHICH IS A FLAMMABLE SOLID AND IS WATER REACTIVE.

IN CASE OF ACCIDENTAL BREAKAGE OR SPILLAGE ONTO SKIN OR CLOTHING, WASH IMMEDIATELY WITH LARGE AMOUNTS OF WATER. DO NOT TAKE INTERNALLY, ALL OF THE CONTENTS ARE POISONOUS.

KEEP OUT OF THE REACH OF CHILDREN

READ MATERIAL SAFETY DATA SHEETS BEFORE PERFORMING TEST.

MANUFACTURER'S WARRANTY

This kit is warranted to be free of defects in material and workmanship until the expiration date stamped on the box. Manufacturer's sole and exclusive liability under this warranty shall be limited to replacement of any kit that is proved to be defective. Manufacturer shall not be liable for any incidental or consequential damages.

Reliable test results are highly dependent upon the care with which the directions are followed and, consequently, cannot be guaranteed.

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Introduction

The HydroScout™ system is designed to accurately and quickly measure the water content in a number of sample matrices. The test uses a specially formulated calcium hydride (CaH₂) reagent to quantitatively convert all the water contained in the sample to hydrogen gas (H₂). The reaction is carried out in a sealed tube and the generated pressure is measured using a specially designed meter. Internal software converts the pressure into water content and the results are displayed directly in the appropriate units.

The HydroScout meter is programed with seven different programs which are easily selected using the program menu. Each program is setup to cover a specific analysis range and matrix combination. Built into each program are all of the conversions necessary to convert the measured pressure into water content. The measurement programs compensate for sample size, dilutions, extractions, reaction efficiencies and conversion to the correct units.

Choosing the Correct Program

The HydroScout meter is programed with seven unique measurement programs. The specific applications for each of the programs are tabulated below. Before beginning any measurements, choose the correct measurement program for the sample matrix and measurement range. Read throughly the specific instructions for the appropriate program to determine what specific reagents and preparation steps are necessary. Selecting the desired program is described below.

Table 1: HydroScout Analysis Program Information

Program	Sample Matrix Types Tested ¹	Units	MDL ³	Maximum	Reagents Needed	Sample Size
A	Used Oil/Liquid Used Oil, Chemtool 250, Paint Waste	Volume Percent	0.15% (v/v)	20% (v/v)	Standard (HS-ORP)	0.25 mL
B	Used Oil/Liquid Used Oil, Chemtool 250, Paint Waste	Volume Percent	5% (v/v)	100% (v/v)	Standard +Dilution Vial (HS-ORP + HS-DVP)	0.25 mL
C	Light Oil/Liquid Exxon Terestic GT32, Mobil DTE Light, Shell Turbo T32, Ideal AW-32, Renolin T-32, Dupont Diamond Class 32, Texaco Havoline 80W-90 Gear Oil	µg/mL	50 µg/mL	10,000 µg/mL	PPM Reagents (HS-LRP)	5 mL
D	Medium Oil/Liquid NAPA Non-Det. SAE 30, Amalie Non-Det. SAE 30, Valvoline Non-Det. SAE 30, Oilzum Non-Det. SAE 50, Gunk Heavy-Duty Brake Fluid, Shell Diala A	µg/mL	50 µg/mL	10,000 µg/mL	PPM Reagents (HS-LRP)	5 mL
E	Heavy Oil/Liquid Exxon Superflo 10W-30, Mobil 1 10W-30, Quaker State Proline 10W-40, Valvoline SAE 50, John Deere Hydraulic Fluid	µg/mL	50 µg/mL	10,000 µg/mL	PPM Reagents (HS-LRP)	5 mL
F	Soil/Solids Sand, Sandy Clay-Loam, Sea Sediment, Topsoil, Georgia Clay, Alabama Clay, Compost, Plant Material	mg H ₂ O	0.25% (w/w)	100% (w/w) ²	Standard +Extraction (HS-SEV)	1-10 grams
G	Paint Paints and Coatings	mg H ₂ O	0.15% (w/w)	100% (w/w) ⁴	Paint Reagents HS-LPT HS-HPT	0.25 mL

¹ For comparison data, see Appendix A.

² Using a 1 gram sample.

³ Method Detection Limit determined using procedure outlined in 40 CFR Ch. 1 Pt. 136, App. B.

⁴ This method has been evaluated by the American Society of Testing and Materials (ASTM) Committee D01 over the range of 2% to 85% water content and is the basis of ASTM Method D 7358.

Programs A and B -> Mid to High Range Water in Oil (EPA Method 9001)

Measurement Range: 0.15% to 100%

Sample Matrix: Used Oil or Liquids

Typical Application: Used oil where moderate to high levels of water are expected.

(Programs A and B are used for measuring water content in an oil/liquid matrix using a 0.25 mL sample.)

Program A covers the range from 0.15% to 20% water content. When an ERROR Code of “Err 4” occurs when testing oil samples under this program, the water content is over 20% (v/v) and the sample must be retested as a diluted sample using Program B. The results for the method is displayed in volume/volume percent.

Program B is used for oil/liquid samples with water concentrations in the 10% to 100% range. If a sample has previously tested to be over 20% (v/v) or is suspected to contain over 20% (v/v) water, it must be run using a dilution solvent vial and Program B. This procedure entails adding an initial 0.25 mL sample to the dilution solvent vial, mixing thoroughly, and introducing a 0.25 mL sample of this mixture into a second sample tube which is part of the basic Reagent package. This sample is then processed under the basic test procedure. The results for the method is displayed in volume/volume percent.

Programs C, D and E -> Low Level Water in Oil

Measurement Range: 50 µg/mL to 10,000 µg/mL

Sample Matrix: Lube Oils, Hydraulic Oils, Brake Fluids, Fuels

Typical Application: Condition monitoring for low levels of water in in-service oils.

(Programs C, D and E are set up to measure low levels of water in an oil/liquid matrix using a 5 mL sample.)

The results for programs C, D and E are reported in µg/mL (displayed as ppm) since the samples sizes are determined by volume. The overall efficiency of the CaH₂ reaction has been determined to be less than 100% with certain oil types due to viscosity and solubility effects. However, since the pressure function is a linear parameter, the meter response value can be corrected to produce accurate results on all oil types. The HydroScout response on low water level oils has been documented for a number of these oil types (See Appendix A). There are three major response groups among the oils tested. The recoveries for the lighter, non-additive, oils such as: turbine oils and diesel fuel are 100% and should be run on program C. Non-detergent motor oils, brake fluids and gear oils average approximately 87% recovery and should be run on program D. Multi-viscosity detergent motor oils and hydraulic fluids with polar additives average 63% recovery and should be run on program E., The results obtained when using programs D and E are automatically corrected to yield accurate results that correlate with Karl Fischer.

NOTE: Phosphate ester-based hydraulic oils and gasoline should be run using Programs A & B only.

If the oil sample is a listed oil type, the corresponding program can be used for analysis. The correct program for oils not listed can be determined by comparing the meter response on program C with a Karl Fischer analysis on the same oil sample. The ratio of the HydroScout result to the Karl Fischer result will determine the correct program to use for future measurements. (NOTE: The HydroScout reports results in volume percent. Be sure to use the same units when comparing HydroScout results to Karl Fischer results.) If the ratio is near 1 (divide the Hydroscout program C result by Karl Fischer result) continue using program C, for ratios between 0.95 and 0.85 use program D and for ratios near 0.6 use program E. (For further assistance in determining the correct program, contact Dexsil.)

Program F -> Water in Soil and Solids

Measurement Range: 0.25% to 100%

Sample Matrix: Soils and Extractable Solids

Typical Application: Moisture content monitoring in agricultural soils.

(Program F should be used to measure moisture content in soils.)

This analysis requires that the soil/solid be extracted using the HydroScout extraction solvent. The extract is then sampled using a standard sampling syringe and analysis procedure.

Program G -> Water in Paint (ASTM Method D 7358)

Measurement Range: 2% – 85%

Sample Matrix: Paints and Coatings

Typical Application: Quality control of paints and coatings.

This method is designed to measure the water content of liquid samples over the range of 1% to 100% (w/w) water content. The reproducibility and repeatability of this method have been evaluated over the range 2% to 85% and are published in ASTM Method D 7358. In order to achieve a result based on the weight of the sample, the sample must be weighed into the reaction vessel. Not only does this allow for a weight based result, but it improves accuracy by eliminating the uncertainty of the volumetric sample size.

If the sample to be analyzed is believed to contain less than 15% water, the sample should be introduced directly into the reaction tube following the procedure for “No Dilution”(See page 10). For samples containing more that 15% water the sample must be diluted and the diluted sample analyzed using the procedure for “with Dilution” (See page 11).

Meter Operation

The HydroScout meter is fully factory calibrated and is ready to operate. Each time the meter is turned on, the micro-processor recalibrates the pressure transducer to zero and performs a QC check on the output voltage. While these checks are being performed, relevant information on the meter will be displayed such as the software version loaded into memory and the current selected measurement program. The meter also has other built-in QC checks to verify measurement integrity (see “Error Codes” below).

Turning the Meter On

Press the <ON> key to turn the meter on. The software version will then be displayed for 5 seconds (-E28,-E30-, etc.) followed by the currently selected measurement program (“A 0-20%”, “b 1-100%”, “c ind”, etc.). When the meter has completed all QC checks, the display reads “tUbE”, indicating that the meter is ready to read a sample (NOTE: It may take a few seconds to zero the sensor and for the display to read “tUbE”).

Making a Measurement

The factory default program is Program A. To make measurements using the default program or the currently selected program: insert the reaction tube into the bottom of the meter until it seats firmly, press the <READ> key and allow the meter to stand upright until the result is displayed. The results are displayed in the units appropriate to the selected program, preceded by the program letter. NOTE: THE METER MUST BE KEPT UPRIGHT WHEN A MEASUREMENT IS MADE. INVERTING THE METER WHILE A TUBE IS INSERTED CAN CAUSE DAMAGE TO THE PRESSURE TRANSDUCER.

Selecting a Program

To select a new program, press the <ON> key once the meter has completed a reading or after the meter has been turned on and has completed all QC checks (display reads “tUbE”). The display will change to the next program in the list after displaying the current program, e.g., “b 1-100%”, indicating that Program B has been selected. Press the <ON> key to scroll through the available programs from A to G followed by “OFF” and then, back to “A 0-20%” again. Press the <READ> key at any point to fix the selection at the displayed program or to turn the meter off when “OFF” is displayed. To begin using the selected program, press the <READ> key and the meter will reset to zero and display “tUbE”. The meter is now ready to read a sample. NOTE: AN ERROR WILL RESULT IF THE METER IS TURNED ON OR THE PROGRAM IS CHANGED WHILE A REACTION TUBE IS IN THE METER.

Turning the Meter Off

The HydroScout meter will turn off automatically after 5 minutes of non-use. To turn off manually, push the <ON> key until the meter displays “OFF” and then push the <READ> key.

Error Codes

The HydroScout performs several QC checks each time the unit is turned on and during each reading to verify that the displayed result is valid. Each time the meter is turned on the pressure transducer output is measured and compared to acceptable values. A high voltage causes an “Err 1” code to be displayed indicating that a tube has been left in the meter while turning it on. (An “Err 1” displayed with no tube in place indicates that the transducer has been damaged.) A low voltage causes an “Err 2” code to be displayed indicating that the pressure transducer has been damaged and must be serviced. If the voltage is acceptable, the meter then sets zero and is ready to take readings by displaying the word “tUbE”. During readings of samples, the pressure is measured a number of times and the trends are analyzed. If the readings are trending upward, the pressure is remeasured and re-analyzed. If this pattern continues, an “Err 3” code is displayed indicating that the reaction is not complete and the reading should be repeated. If the pressure is trending downward, an “Err 5” code is displayed indicating that the tube is leaking and that the sample testing should be re-run from the start. Once a reading has stabilized, the result is calculated and displayed. If the pressure is outside of the linear range, an “Err 4” code will be displayed indicating that the sample should be rerun using a smaller sample size or a dilution technique and a different program. (See Table 1)

Table 2: Error Codes

Error Code	Explanation	Likely Cause	Remedy
Err 1	Zero voltage too high.	Reaction tube in place when turning meter on or switching programs.	Remove tube and re-select program.
Err 2	Zero voltage too low.	Excessive drift in transducer output caused by damage.	Call for service.
Err 3	Pressure readings trending up.	Reaction has not gone to completion.	Wait another minute and re-read.
Err 4	Reading outside of linear range.	Water content greater than 20% (v/v).	Re-run sample using dilution vials and "Program B".
Err 5	Pressure too low during sample reading.	Leaking tube or cap. Tube squeezed while inserting gray stopper.	Re-run sample using new reaction tube. If problem persists call for assistance.
Lo Pr	Low Battery	Low Battery	Remove four screws. Replace battery with 9V alkaline or lithium only.

Interferences

In addition to the testing the effectiveness of the HydroScout system in a number of matrices, extensive testing has also been conducted to document possible interferences and/or cross reactivities due to additives or components in a sample matrix. The following compounds have been found to produce no measurable response to the HydroScout system and, therefore, cause no interference at levels up to 40%:

Ethanol
Methanol
Acetone*
Methyl ethyl ketone
Tetrahydrofuran
Diethylene glycol dimethyl ether
Ethylene glycol*
Diethylene glycol

Dipropylene glycol
Stearic acid
2-Ethyl hexanoic acid
Lead oxide (II and III)
Aluminum oxide (Brockman I)

*0.25 mL sample size program A and B

When using the 5 mL sample size programs, i.e., programs C, D, or E, ethylene glycol and acids have been found to produce a positive result. Ethylene glycol responds at approximately 7% of the volumetric equivalent.

Step by Step Instructions for Meter Operation

- Step 1. Press the <ON> Key.
SCREEN: “-E34- ” is displayed followed by the currently selected program (i.e. “A 0-20%”) , then flashes “-bL-”, and then “tUbE.”
(“-E34-” is the current software version and “-bL-” indicates the instrument is undergoing internal calibration.)
- Step 2. If Program “A” is not the desired program, press the <ON> key to change it by following the procedure described in “Selecting a Program”.
- Step 3. ***With the meter held upright:*** Insert the reaction tube into the bottom of the meter until it seats firmly.
- Step 4. Press the <READ> Key.
SCREEN: “CALC” flashes,
then:
SCREEN: “A XXXX ”

(Where “A” is the letter designating the current program and “XXXX” is the reading in the units appropriate for the current program.)
- Step 5. Record result, remove reaction tube and insert new sample.
- Step 6. Press the <READ> key.
- Step 7. Repeat steps 3-6 until all samples have been read.
- Step 8. Turn off meter manually or meter will turn off automatically after 5 minutes of non-use.

NOTE: Meter is off when screen is blank.

Analysis Instructions for Each Sample Type and Analysis Program

Program A → Procedure for Testing Water Content up to 20%

(Reagent System: HS-ORP)

1. **PREPARATION:** Remove a sampling syringe and a white-capped reaction tube from the reagent box. Check the contents to ensure that all items are present and intact. Remove the white cap and black septum from the reaction tube. Carefully remove a green water ampule from the cardboard carton and insert it fully into the top of the protective ampule sleeve containing the grayish white ampule. Return the protective ampule sleeve to the reaction tube with the grayish white ampule on the bottom and place the tube into the reagent box.
2. **SAMPLE INTRODUCTION:** Work the plunger of the empty sampling syringe a few times to ensure that it slides easily. Place the tip of the syringe into the oil sample to be tested (sample must be well mixed and representative) and slowly pull back on the plunger until it reaches the stop and cannot be pulled further. When drawing up the sample, make sure there are no air bubbles in the syringe. If not free of air bubbles, depress the plunger again and draw a second sample slowly. Remove the syringe from the oil sample and wipe any excess oil from the outside of the syringe with a tissue wipe. Insert the sampling syringe into the reaction tube and dispense the oil sample by depressing the plunger. Replace the black septum (hole on top) by pressing down on the septum with your thumb while gripping the reaction tube firmly using a uniform pressure along the side of the tube and supporting the bottom. (NOTE: Squeezing the tube to cause a deformation in the sides of the tube will result in an artificially low result.) Replace the white cap TIGHTLY.
3. **REACTION:** Break the bottom (grayish white) ampule in the reaction tube by compressing the sides of the tube. Mix thoroughly by shaking the reaction tube vigorously for 30 seconds. Allow the reaction to proceed for a total of 2 minutes. DO NOT BREAK THE GREEN AMPULE.
4. **ANALYSIS:** Press the <ON> key and make sure the meter is set for “A 0-20%” (NOTE: If changing the program, see “Selecting a Program” in the manual). Once the meter displays “tUbE”, insert the reaction tube into the opening at the bottom of the meter. The hole in the white cap must be lined up with the metal pin extending from the inside of the hole of the meter. Push the reaction tube all the way into the meter. Press the <READ> key on the face of the meter. The display will flash the word “CALC,” and the results will be displayed in volume percent (v/v%) Record the results. KEEP THE METER AND REACTION TUBE UPRIGHT AT ALL TIMES.

5. **DISPOSAL:** Carefully remove the reaction tube from the meter. Vent the reaction tube by inserting it completely into the specially designed venting cap unit and remove slowly. REACTION TUBE AND CAP MUST BE UPRIGHT AND POINTED AWAY FROM USER AND BYSTANDERS. Tap the reaction tube on a hard surface to shake any loose glass to the bottom of the tube, then break the top (green) ampule in the reaction tube by compressing the sides of the reaction tube. Shake the tube for 15 seconds. After 5 minutes, vent the tube again. Discard the reaction tube as laboratory waste. DO NOT REMOVE THE WHITE CAP

Program B → Procedure for Testing Water Content up to 100%

(Reagent Systems: HS-ORP + HS-DVP-12)

1. **PREPARATION:** Remove a screw-capped dilution vial from the fluted cardboard carton, a sampling syringe and a white-capped reaction tube from the reagent box. Check the contents to ensure that all items are present and intact. Remove the white cap and black septum from the reaction tube. Carefully remove a green water ampule from the cardboard carton and insert it fully into the top of the protective ampule sleeve containing the grayish white ampule. Return the protective ampule sleeve to the reaction tube with the grayish white ampule on the bottom and place the reaction tube into the reagent box.
2. **SAMPLE INTRODUCTION:** Work the plunger of the empty sampling syringe a few times to ensure that it slides easily. Place the tip of the syringe into the oil sample to be tested (sample must be well mixed and representative) and slowly pull back on the plunger until it reaches the stop and cannot be pulled further. When drawing up the sample, make sure there are no air bubbles in the syringe. If not free of air bubbles, depress the plunger again and draw a second sample slowly. Remove the syringe from the oil sample and wipe any excess oil from the outside of the syringe with a tissue wipe.
3. **SAMPLE DILUTION:** Remove the screw-cap from the dilution vial. Insert the sampling syringe into the dilution vial and dispense the oil sample by depressing the plunger. Proceed to mix the solution thoroughly by repeated drawing and re-dispensing of the solution into the dilution vial with the syringe (3 or 4 times). Using the same sampling syringe, draw up a sample from the diluted solution wiping any excess liquid from the outside of the syringe. Dispense the sample into the reaction tube. Replace the black septum (hole on top) by pressing down on the septum with your thumb while gripping the reaction tube firmly using a uniform pressure along the side of the tube and supporting the bottom. (NOTE: Squeezing the tube to cause a deformation in the sides of the tube will result in an artificially low result.) Replace the white cap TIGHTLY.
4. **REACTION:** Break the bottom (grayish white) ampule in the reaction tube by compressing the sides of the tube. Mix thoroughly by shaking the reaction tube vigorously for 30 seconds. Allow the reaction to proceed for a total of 2 minutes. DO NOT BREAK THE GREEN AMPULE.
5. **ANALYSIS:** Press the <ON> key and make sure the meter is set for “b 1-100%” (NOTE: If changing the program, see “Selecting a Program” in the manual). Once the meter displays “tUbE”, insert the reaction tube into the opening at the bottom of the meter. The hole in the white cap must be lined up with the metal pin extending from the inside of the hole of the meter. Push the reaction tube all the way into the meter. Press the <READ> key on the face of the meter. The display will flash the word “CALC,” and the results will be displayed in volume percent (v/v%). Record the results. KEEP THE METER AND REACTION TUBE UPRIGHT AT ALL TIMES.
6. **DISPOSAL:** Carefully remove the reaction tube from the meter. Vent the reaction tube by inserting it completely into the specially designed venting cap unit and remove slowly. REACTION TUBE AND CAP MUST BE UPRIGHT AND POINTED AWAY FROM USER AND BYSTANDERS. Tap the reaction tube on a hard surface to shake any loose glass to the bottom of the tube, then break the top (green) ampule in the reaction tube by compressing the sides of the reaction tube. Shake the tube for 15 seconds. After 5 minutes, vent the tube again. Discard the reaction tube and dilution vial as laboratory waste. DO NOT REMOVE WHITE CAP.

Programs C, D & E → Procedure for Testing Low Levels of Water in Oil Using a 5 mL Sample Size

(Reagent System: HS-LRP)

WARNING - DO NOT USE 5 ML SAMPLES OF PHOSPHATE ESTER BASED HYDRAULIC FLUIDS, GASOLINE OR SOLVENTS SUCH AS ACETONE. THESE MATERIALS WEAKEN THE REACTION TUBES WHICH MAY RESULT IN TUBE FAILURE.

1. **PREPARATION:** Remove a sampling syringe and a white-capped reaction tube from the reagent box. Check the contents to ensure that all items are present and intact. Remove the white cap and black septum from the reaction tube. Carefully remove a green water ampule from the cardboard carton and insert it fully into the top of the protective ampule sleeve containing the grayish white ampule. **Do not replace the protective ampule sleeve into the tube at this time.**
2. **SAMPLING:** Using a clean polyethylene sampling pipette, pipette 5 mL of the oil to be tested into the reaction tube, e.g., up to 5 mL line marked on side of the reaction tube (**NOTE: The protective ampule sleeve is not to be in the tube at this time.**). Insert the protective ampule sleeve into the reaction tube with the grayish white ampule on the bottom. Replace the black septum (hole on top) by pressing down on the septum with your thumb while gripping the reaction tube firmly using a uniform pressure along the side of the tube and supporting the bottom. (**NOTE: Squeezing the tube to cause a deformation in the sides of the tube will result in an artificially low result.**) Replace the white cap **TIGHTLY**. **NOTE: A homogeneous, representative sample of the oil being tested must be used to obtain accurate results.**
3. **REACTION:** Break the bottom (grayish white) ampule in the reaction tube by compressing the sides of the tube. Mix thoroughly by shaking the reaction tube vigorously for 30 seconds. Allow the reaction to proceed for a total of 4 minutes. The mixture will bubble and perhaps foam during this time **DO NOT BREAK THE GREEN AMPULE**. (**NOTE: Some oils require a longer reaction time to achieve complete reaction. This is especially true for oils run on program C. If this is the case, shake the reaction tube again for 30 seconds and let stand for an additional 4 minutes.**)
4. **ANALYSIS:** Press the <ON> key once to activate the meter. Ensure the meter is set for the correct program C, D or E for the sample matrix being tested (See "Selecting a Program" for instructions on how to change programs.). Once the meter displays "tUbE", insert the reaction tube into the opening at the bottom of the meter. The hole in the white cap must be lined up with the metal pin extending from the inside of the hole of the meter. Push the reaction tube all the way into the meter. Press the <READ> key on the face of the meter. The display will flash the word "CALC," and the results will be in **micrograms of water per milliliter of sample displayed as ppm**. Record the results. **KEEP THE METER AND REACTION TUBE UPRIGHT AT ALL TIMES.**
5. **DISPOSAL:** Carefully remove the reaction tube from the meter. Vent the reaction tube by inserting it completely into the specially designed venting cap unit and remove slowly. **REACTION TUBE AND CAP MUST BE UPRIGHT AND POINTED AWAY FROM USER AND BYSTANDERS.** Tap the reaction tube on a hard surface to shake any loose glass to the bottom of the tube, then break the top (green) ampule in the reaction tube by compressing the sides of the reaction tube. Shake the tube for 15 seconds. After 5 minutes, vent the tube again. Discard the reaction tube as laboratory waste. **DO NOT REMOVE WHITE CAP.**
6. **CALCULATION:** The meter is programed to report the result in **micrograms per milliliter and displayed as ppm**. If a result is to be reported as a weight/weight value, divide the HydroScout result by the density of the oil sample.

$$\text{Water in Oil } (\mu\text{g/g}) = R/d$$

where:

R = Meter reading in $\mu\text{g/mL}$

d = Density of oil in g/mL

Program F → Procedure for the Analysis of Water in Soils and Solids

(Reagent Systems: HS-SEV)

1. **PREPARATION:** Remove a sampling syringe and a white-capped reaction tube from the reagent box and the extraction solvent ampule from the colored-capped soil extraction tube. Check the contents to ensure that all items are present and intact. Remove the white cap and black septum from the reaction tube. Carefully remove a green water ampule from the cardboard carton and insert it fully into the top of the protective ampule sleeve containing the grayish white ampule. Return the protective ampule sleeve to the reaction tube with the grayish white ampule on the bottom, place the reaction tube into the reagent box.
2. **SAMPLE EXTRACTION:** Remove the colored cap from the soil extraction tube and tare on an electronic scale. Weigh 5-10 grams of soil to be tested into the soil extraction tube and record the weight. Add the contents of one break-top ampule of extraction solvent (white polypropylene top) to the soil extraction tube, re-cap and shake for 1 minute. Allow soil in the soil extraction tube to settle for 2 minutes.
3. **SAMPLING EXTRACTED SAMPLE:** Work the plunger of the empty sampling syringe a few times to ensure that it slides easily. After soil has settled, draw up a sample from the extract layer with the sampling syringe ensuring that there are no air bubbles in the syringe. If not free of air bubbles, depress the plunger again and draw a second sample slowly. Wipe any excess solvent from the outside of the syringe with a tissue wipe and dispense the sample into the reaction tube. Replace the black septum (hole on top) by pressing down on the septum with your thumb while gripping the reaction tube firmly using a uniform pressure along the side of the tube and supporting the bottom. (NOTE: Squeezing the tube to cause a deformation in the sides of the tube will result in an artificially low result.) Replace the white cap TIGHTLY.
4. **REACTION:** Break the bottom (grayish white) ampule in the reaction tube by compressing the sides of the tube. Mix thoroughly by shaking the reaction tube vigorously for 30 seconds. The mixture will bubble and perhaps foam during this time. Allow the reaction to proceed for 1.5 minutes. Shake the reaction tube again for 30 seconds and let stand for an additional 30 seconds. DO NOT BREAK THE GREEN AMPULE
5. **ANALYSIS:** Press the <ON> key once to activate the meter. Ensure the meter is set for the “F Soil^{mg}.” program for the sample matrix being tested (See “Selecting a Program” for instructions on how to change programs.). Once the meter displays “tUbE”, insert the reaction tube into the opening at the bottom of the meter. The hole in the white cap must be lined up with the metal pin extending from the inside of the hole of the meter. Push the reaction tube all the way into the meter. Press the <READ> key on the face of the meter. The display will flash the word “CALC,” and the results will be displayed in milligrams of water in the sample. Record the results. KEEP THE METER AND REACTION TUBE UPRIGHT AT ALL TIMES.
6. **DISPOSAL:** Carefully remove the reaction tube from the meter. Vent the reaction tube by inserting it completely into the specially designed venting cap unit and remove slowly. REACTION TUBE AND CAP MUST BE UPRIGHT AND POINTED AWAY FROM USER AND BYSTANDERS. Tap the reaction tube on a hard surface to shake any loose glass to the bottom of the tube, then break the top (green) ampule in the reaction tube by compressing the sides of the tube. Shake the tube for 15 seconds. After 5 minutes, vent the tube again. Discard the reaction tube and soil extraction tube as laboratory waste. DO NOT REMOVE WHITE CAP
7. **CALCULATION:** The meter is programed to display the weight of the water, in **milligrams**, contained in the sample. Calculate the water content in weight percent using the following equation:

$$\text{Water in Soil, wt. percent} = R/(10S_1)$$

where:

R = Meter reading in milligrams of water
S₁ = Weight of soil sample, in grams

Procedure G → Procedure for the Analysis of Paints and Coatings

(Reagent System: HS-LPT)

Procedure for Expected Water Content Less than 15% (No Dilution)

1. **PREPARATION:** Remove a sampling syringe and a white-capped tube from the box. Check the contents to ensure that all items are present and intact. Remove the white cap and black septum from the reaction tube. Insert one green water ampule into the top of the polypropylene ampule sleeve containing the grayish-white ampule and replace the ampule sleeve into the reaction tube with the grayish-white ampule on the bottom
2. **SAMPLE INTRODUCTION:** Remove the cap from the sampling syringe. Work the plunger on the empty sampling syringe a few times to ensure that it slides easily. Place the tip of the syringe into the paint sample to be tested and slowly pull back on the plunger until it reaches the stop and cannot be pulled further. Remove the syringe from the paint sample and wipe any excess paint from the outside of the syringe with a tissue wipe. Place a syringe cap on the syringe and weigh; record the weight (gross sample weight) to the nearest 0.1 mg. Dispense this sample into the reaction tube. Replace the syringe cap and reweigh the syringe and record the weight (sample tare weight). The difference in the two weights becomes the sample weight S_1 to be used in equation 1 below. Replace the black septum (hole on top) by gripping the tube firmly and pressing the septum down with the thumb. (NOTE: Do not squeeze the sides of the tub while inserting the septum as this will cause a low reading.) Replace the white cap **TIGHTLY**, turning it until it cannot be turned further.
3. **REACTION:** Break the bottom (gray) ampule in the tube by compressing the sides of the tube. Mix thoroughly by shaking the tube vigorously for 30 seconds. The mixture will be seen to bubble and perhaps foam. Allow the reaction to proceed for 2 minutes. Shake the reaction tube again for 10 seconds and let stand for an additional 1 minute.
4. **ANALYSIS:** Press the <ON> key to activate the meter. Ensure the meter is set for "Program G" (See "Selecting a Program" in the manual on how to change programs). Once the meter displays "tUbe", insert the reaction tube firmly into the opening at the bottom of the meter. The hole in the white cap must be lined up with the metal pin extending from the inside of the hole in the meter. Push the reaction tube all the way into the meter. Press the <READ> key on the face of the meter. The display will flash the word "CALC," then the results will displayed in **milligrams** of water in the sample. Record the results. **KEEP THE METER AND TUBE UPRIGHT AT ALL TIMES.**
5. **DISPOSAL:** Carefully remove the reaction tube from the meter. Vent tube by inserting completely into venting cap unit and remove slowly. **REACTION TUBE AND CAP MUST BE UPRIGHT AND POINTED AWAY FROM USER AND BYSTANDERS.** Tap the tube on the bench top to shake any loose glass to the bottom of the tube, then break the top (green) ampule in the reaction tube by compressing the sides of the tube. Shake the tube for 15 seconds. After 5 minutes, vent the tube again. Discard the reaction tube and vial as laboratory waste. **DO NOT REMOVE WHITE CAP.**
6. **CALCULATION:** The meter is programed to display the weight, in **milligrams**, of the water contained in the sample introduced into the reaction tube (In this case the raw sample). Calculate the water content in the sample in weight percent using the following equation:

$$\text{Water in paint, wt. percent} = R/(S_1 \times 10) \quad (1)$$

where:

R = Meter reading in **milligrams** of water

S_1 = actual weight of whole paint sample, in **grams**

Procedure G → Procedure for the Analysis of Paints and Coatings (cont'd)

(Reagent System: HS-HPT)

Procedure for Expected Water Content Greater than 15% (with Dilution)

1. **PREPARATION:** Remove a dilution vial, two sampling syringes, two caps and a white-capped tube from the box. Check the contents to ensure that all items are present and intact. Remove the white cap and black septum from the reaction tube and the cap from the dilution vial. Insert one green water ampule into the top of the polypropylene ampule sleeve and replace the ampule sleeve into the reaction tube with the grayish-white ampule on the bottom.
2. **SAMPLE INTRODUCTION:** Remove the cap from an empty sampling syringe. Work the plunger on the empty sampling syringe a few times to ensure that it slides easily. Place the tip of the syringe into the paint sample to be tested and slowly pull back on the plunger until it reaches the stop and cannot be pulled further. Remove the syringe from the paint sample and wipe any excess paint from the outside of the syringe with a tissue wipe. Place a syringe cap on the syringe and weigh; record the weight (gross sample weight) to the nearest 0.1 mg.
3. **SAMPLE DILUTION:** Remove the syringe cap and insert the sampling syringe into the dilution vial and dispense the paint sample by depressing the plunger. (Do not allow the syringe to contact the dilution solvent when dispensing the sample.) Replace the syringe cap and reweigh the syringe and record the weight (sample tare weight). The difference in the two weights becomes the sample weight S_1 to be used in equation 2 below. Replace the white cap on the dilution vial and shake the vial vigorously for at least 10 seconds. Remove the vial cap and quickly, using a new sampling syringe, draw up a sample from the solution. Wipe any excess solution from the outside of the syringe, cap with a fresh cap and weigh the syringe. Record this weight (diluted sample gross weight). Dispense this sample into the reaction tube. Replace the cap and reweigh the syringe and record this weight (diluted sample tare weight). The difference in the two weights becomes the sample weight S_2 to be used in equation 2 below. Replace the black septum (hole on top) by gripping the tube firmly and pressing the septum down with the thumb. (NOTE: Do not squeeze the sides of the tube while inserting the septum as this will cause a low reading.) Replace the white cap **TIGHTLY**, turning it until it cannot be turned further.
4. **REACTION:** Break the bottom (gray) ampule in the tube by compressing the sides of the tube. Mix thoroughly by shaking the tube vigorously for 30 seconds. The mixture will be seen to bubble and perhaps foam. Allow the reaction to proceed for 2 minutes. Shake the reaction tube again for 10 seconds and let stand for an additional 1 minute.
5. **ANALYSIS:** Press the <ON> key to activate the meter. Ensure the meter is set for "Program G" (See "Selecting a Program" in the manual on how to change programs). Once the meter displays "tUBE", insert the reaction tube firmly into the opening at the bottom of the meter. The hole in the white cap must be lined up with the metal pin extending from the inside of the hole in the meter. Push the reaction tube all the way into the meter. Press the <READ> key on the face of the meter. The display will flash the word "CALC," then the results will displayed in **milligrams** of water in the diluted sample. Record the results. **KEEP THE METER AND TUBE UPRIGHT AT ALL TIMES.**
6. **DISPOSAL:** Carefully remove the reaction tube from the meter. Vent tube by inserting completely into venting cap and remove slowly. **REACTION TUBE AND CAP MUST BE UPRIGHT AND POINTED AWAY FROM USER AND BYSTANDERS.** Tap the tube on the bench top to shake any loose glass to the bottom of the tube, then break the top (green) ampule in the reaction tube by compressing the sides of the tube. Shake the tube for 15 seconds. After 5 minutes, vent the tube again. Discard the tube and vial as laboratory waste. **DO NOT REMOVE WHITE CAP.**
7. **CALCULATION:** The meter is programed to display the weight, in **milligrams**, of the water contained in the sample introduced into the reaction tube (In this case the diluted sample). Calculate the water content in the original paint sample in weight percent using the following equation:

$$\text{Water in paint, wt. percent} = (R/S_2)(1+(2.15/S_1))/10 \quad (2)$$

where:

R = Meter reading in **milligrams** of water

S_1 = actual weight of whole paint sample, in **grams**

S_2 = actual weight of diluted paint sample, in **grams**

2.15 = Weight of the dilution solvent, in **grams**

Appendix A: Comparison Data

Determination of Water in Used Oil on Program A and B, w/w%

Expected	HydroScout ¹	Karl Fischer
0	0.161	0.061
0.1	0.148	0.145
0.2	0.226	0.255
0.5	0.458	0.560
1.0	0.948	1.07
2.0	2.36	2.46
5.0	5.03	5.05
10.0	9.82	9.99
20.0	20.2	20.0
25.0	26.37	26.05
50.0	50.01	50.61

¹ HydroScout results converted to w% using sample density

Analysis of Used Oil Certified Reference Materials on Programs A and B²

CRM	Certified Value, wt %	HydroScout ¹ , wt %	Karl Fischer, wt %
ERM-34	1.95	1.92±0.02	1.86±0.09
ERM-35	5.86	5.91±0.61	5.82±0.79
ERM-36	10.3	10.30±0.53	10.3±0.43
ERM-41	87.4	88.4±6.7	86.4±6.6

¹ HydroScout results converted to w% using sample density

² ERM-34 to 41 Water Content in Used Oil Mixtures from Environmental Reference Materials, Inc.

Analysis of Low Levels of Water in Turbine and Gear Oils Using Program C

Oil Type	Karl Fischer ($\mu\text{g/mL}$)	HydroScout ($\mu\text{g/mL}$)	C.V.
Exxon Teresstic Turbine Oil GT32	18.7	0	.169
	200	140	.124
	396	404	.023
	793	825	.019
Mobil DTE Turbine Oil Light	26	53	.08
	236	202	.007
	448	410	.043
	838	778	.026
Shell Turbo T32	23	34	.019
	220	223	.04
	433	403	.01
	825	800	.038
Ideal Turbine Oil AW-32	39	43	.072
	210	208	.013
	400	435	.005
	759	842	.013
Renolin Turbine Oil T-32	28	40	.067
	250	238	.024
	409	410	.039
	798	832	.01
Dupont Diamond Class Turbine Oil 32	16	117	.056
	202	220	.048
	429	399	.03
	833	783	.011
Texaco Havoline 80W-90 Gear Oil	285	250	.062
	510	460	.024
	721	659	.011
	1120	1037	.008

Analysis of Water Content in Various Fluids Using Program D

Fluid Type	Karl Fischer ($\mu\text{g/mL}$)	HydroScout ($\mu\text{g/mL}$)	C.V.
NAPA Non-Detergent SAE 30	0	17	.073
	88	117	.069
	176	217	.08
	351	303	.071
	702	661	.064
	1405	1450	.048
Amalie Non-Detergent SAE 30	0	2	.051
	87	101	.038
	175	166	.057
	349	374	.009
	696	712	.027
	1397	1528	.015
Valvoline Non-Detergent SAE 30	0	30	.059
	88	96	.042
	175	136	.061
	350	368	.028
	701	784	.02
	1401	1497	.041
Oilzum Non-Detergent SAE 50	0	76	.051
	89	126	.024
	177	212	.06
	355	404	.02
	707	799	.009
	1419	1595	.016
Gunk Heavy-Duty Brake Fluid	3835	4123	.01
	4318	4323	.013
	4586	4795	.009
	5136	5341	.002
Shell Diala A	0	70	.039
	88	121	.054
	177	189	.055
	353	372	.012
	707	715	.041
	1413	1506	.018

Analysis of Various Fluids Using Program E

Fluid Type	Karl Fischer ($\mu\text{g/mL}$)	HydroScout ($\mu\text{g/mL}$)	C.V.
Exxon Superflo 10W-30	0	82	.082
	87	150	.014
	174	213	.029
	348	395	.009
	695	632	.011
	1390	1425	.037
Mobil 1 10W-30	0	36	.044
	86	95	.035
	172	126	.047
	343	289	.035
	687	602	.039
	1374	1313	.015
Quaker State SAE 30	0	7	.018
	88	105	.06
	176	183	.104
	352	333	.044
	703	597	.04
	1406	1491	.018
Quaker State Proline 10W-40 (Used)	0	26	.079
	89	195	.076
	178	294	.026
	357	409	.023
	714	801	.010
	1428	1370	.027
Valvoline SAE 50	0	11	.041
	88	87	.047
	177	129	.039
	354	284	.035
	708	632	.064
	1415	1482	.021
John Deere Hydraulic Fluid	0	0	.03
	88	145	.03
	176	233	.011
	352	330	.042
	704	664	.037
	1409	1597	.013

Analysis of Various Soil Types Using Program F

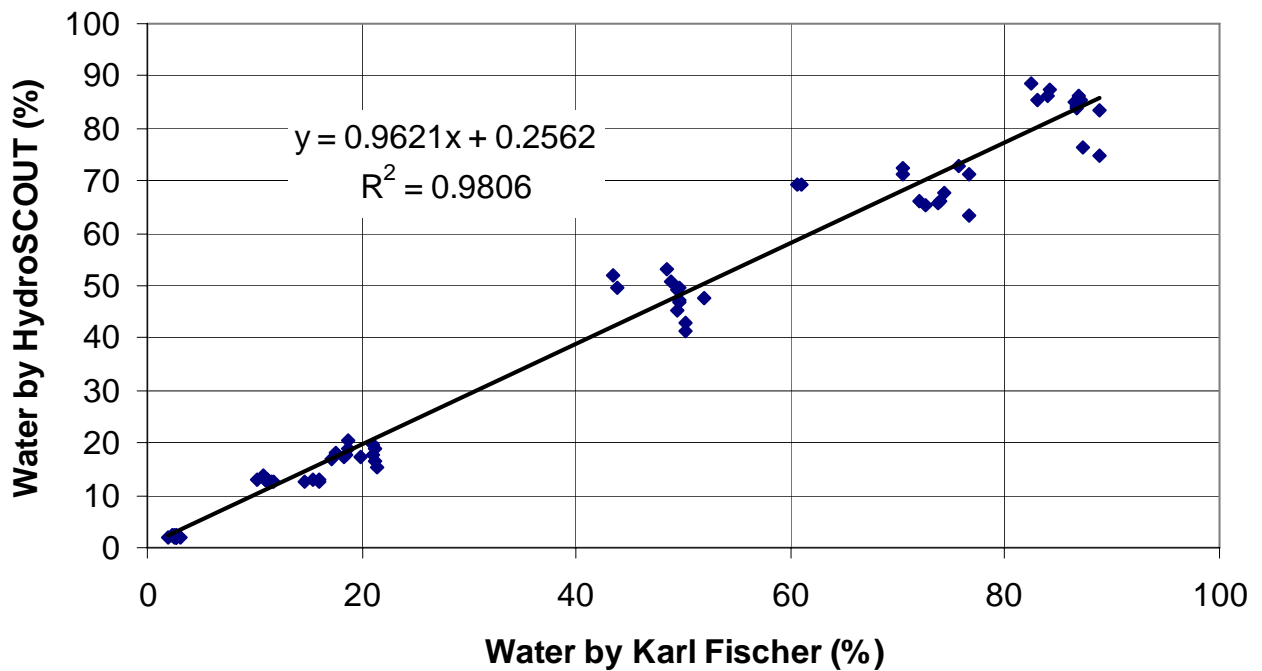
Soil Type / Spiked Water Content (wt %)	Average HydroScout Result (wt %)	C.V.
Composite Sandy-Clay-Loam		
0	0	-
5.7	6.19	0.0219
10.1	10.65	0.0387
20.1	19.79	0.0236
Sea Sediment		
0	0.08	-
10.2	9.66	0.0131
20.0	18.68	0.0184
29.9	26.63	0.0081
Topsoil		
0	0.21	-
5.0	5.13	0.0377
10.1	10.33	0.0248
20.0	19.50	0.0026
Sand		
0	0.22	-
5.1	5.30	0.0266
10.1	10.30	0.0274
15.0	15.42	0.0304
Clay Loam I		
0	0	-
5.0	4.47	0.0354
10.2	10.50	0.0032
20.1	18.92	0.0041
Clay Loam II		
0	0	-
5.2	5.49	0.0104
10.1	10.19	0.0065
20.1	19.96	0.0038
Georgia Clay		
0	0	-
5.1	5.23	0.0168
10.0	10.17	0.0231
20.6	20.29	0.0281
Alabama Clay		
0	0	-
5.0	4.87	0.0259
10.1	10.14	0.0028
21.2	20.21	0.0030

Analysis of Various Coatings Using Program G

Data from the ASTM Inter-Laboratory Study of the Calcium Hydride Method D7358 comparing the HydroScout results with Karl Fischer. The coatings analyzed were:

- Fluid A: Clear Acrylic sealer
- Fluid B: Acrylic white tint base
- Fluid C: Acrylic stain
- Fluid D: Phenolic resin containing approximately 10% unreacted Phenol.
- Fluid E: Pigmented polyester polyol resin system containing MEK, Toluene, Ethyl Acetate, PM Acetate, and Ethyl 3-Ethoxy Propionate.
- Fluid F: Phenolic coating containing approximately 5% unreacted Phenol as well as Ethyl Alcohol, PM Acetate, and Dipropylene Glycol Monomethyl Ether.

Calcium Hydride vs Karl Fischer



Appendix B: Meter Specifications

A/D Resolution:	12 bit																		
Measurement Range:	Program and analyte dependant																		
	<table><thead><tr><th>Program</th><th>MDL</th><th>Max Linear Range</th></tr></thead><tbody><tr><td>A</td><td>0.15% (v/v)</td><td>20% (v/v)</td></tr><tr><td>B</td><td>5% (v/v)</td><td>100% (v/v)</td></tr><tr><td>C, D& E</td><td>50 ug/mL</td><td>10,000 ug/mL</td></tr><tr><td>F</td><td>0.25% (wt/wt)*</td><td>100% (wt/wt)**</td></tr><tr><td>G</td><td>0.15% (wt/wt)</td><td>100% (wt/wt)</td></tr></tbody></table>	Program	MDL	Max Linear Range	A	0.15% (v/v)	20% (v/v)	B	5% (v/v)	100% (v/v)	C, D& E	50 ug/mL	10,000 ug/mL	F	0.25% (wt/wt)*	100% (wt/wt)**	G	0.15% (wt/wt)	100% (wt/wt)
Program	MDL	Max Linear Range																	
A	0.15% (v/v)	20% (v/v)																	
B	5% (v/v)	100% (v/v)																	
C, D& E	50 ug/mL	10,000 ug/mL																	
F	0.25% (wt/wt)*	100% (wt/wt)**																	
G	0.15% (wt/wt)	100% (wt/wt)																	
	* Using a 10 gram sample.																		
	** Using a 1 gram sample.																		
Program Storage:	Non-Volatile																		
Display :	4 Digit 0.35 inch LCD with Program Indicator																		
Battery:	One 9V Lithium (included) [Use only Alkaline or Lithium]																		
Battery Life:	10,000 measurements or 10 year shelf-life (using 1200 mAh lithium battery)																		
Operating Temperature:	4°C to 40°C																		
Dimensions:	length - 5.9" (150 mm), width - 3.2" (81 mm), height - 1.8" (46 mm)																		
Weight:	9.2 oz (261 g)																		