DIAGNOSTIC TEST FOR WATER IN OIL

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Moisture in oil is an important parameter that must be addressed when considering contaminates in lubricating oils. From the simple non-quantitative “crackle tests” to the sophisticated Karl Fisher method, thousands of water in oil tests are performed annually. Most of the methods used today are performed in the laboratory by trained analytical chemists and technicians. Real time results are not practical and the turn around time for most analysis can be as long as two weeks. A real time method that is both easy to run and cost effective has been developed and is marketed by Dexsil Corporation.

HydroScout was originally designed for quantifying percent levels of water in used oil destined for recycling. By increasing the sample size and modifying the chemistry, this new test method has a method detection limit (MDL) of 50 ppm. The benefits of this quantitative method are: its field portability for real time results, ease of use with relatively no set up time, pre-calibrated instrument, pre-standardized and environmentally safe reagents.

The new method is based on the standard reaction of water with calcium hydride to produce one mole of hydrogen for every mole of water.

$$CaH_2 + 2H_2O \rightarrow Ca(OH)_2 + 2H_2 \uparrow$$

By encapsulating the calcium hydride in crushable glass ampules and reacting the oil in a sealed, soft-sided tube, all reagents can be pre-measured and all supplies are disposable. The reaction tube is sealed with a rubber septum which, when inserted into the HydroScout meter, is punctured to allow the hydrogen pressure to be measured directly using a microprocessor controlled pressure transducer. Using the ideal gas law, the internal pressure of the reaction tube is converted into the amount of water in the sample. Depending on the program chosen, stored constants are used to calculate the water content in the oil.

To verify the effectiveness of this method in reacting all of the water in an oil sample, six different turbine oils were spiked with water at various levels, reacted and the pressure measured. The actual water content of the oils was determined using a Karl Fischer method using azeotropic distillation. Figure 1 shows the measured pressure plotted versus the water content in the oil for all six oils. The dotted line is the pressure predicted from the ideal gas law and the reaction stoichiometry. The solid line is the best fit line calculated from the regression analysis of the data. The good agreement with theory and the linearity of the pressure as a function of water content, indicate complete recovery over the range of water concentrations tested.

Analyzing these oils using the standard oil analysis program illustrates the accuracy in predicting the oil content based on the measured pressure for turbine oils. (See Figure 2) The solid line is the theoretical result predicted from the Karl Fischer result: note that the R^2 from the regression analysis was 0.98.

To achieve complete reaction, all of the water present must come into contact with the calcium hydride. Because the water is suspended in a generally non-polar matrix, it might be expected that the nature of the oil being tested will have an effect on the ability of the calcium hydride to come in contact with all of the water and hence on the accuracy of the HydroScout result. Among the matrix parameters expected to affect the results would be the viscosity of the base stock and the polarity of the additives used to formulate the oil. The viscosity of the oil would tend to physically prevent the oil from mixing with the calcium hydride whereas the additives, being generally more polar than the base stock, would tend to segregate the water from the reactants by a chemical attraction to the water itself.

To investigate these effects, various oils and hydrocarbon based fluids (non-detergent and detergent motor oils, single and multi-viscosity motor oils, gear oil, brake fluid and hydraulic oils) were spiked at different levels, measured using the new procedure and the results compared to Karl Fisher. Each of the spiked oils/fluids produced a linear response with good correlation to the Karl Fischer results.

The oils/fluids tested tended to fall into three groups, apparently based on the affinity of the fluid for water. The lighter turbine oils and fuel oils as well as the more viscous gear oil produced nearly theoretical pressure readings indicating that the water was reacted completely. A second group was identified with approximately an 85% to 90% recovery, comprised of the single viscosity, non-detergent motor oils, brake fluid and mineral oil dielectric fluids. (See Figure 3) A third group was also evident, with approximately a 60% to 65% recovery. This group includes the multi-viscosity motor oils, detergent motor oils and hydraulic oils. (See Figure 4) Other fluids tested such as, brake fluid, gear oil and mineral oil dielectric fluid fall on one of the preprogramed response curves. (See Figure 5)

Programming the meter with three different conversion programs allows for the accurate determination of low levels of water in all three groups of oils. For the specific oil/fluid types tested here, the correct response factors have been determined. For other types, a single comparison point can be used to determine the correct program to use.

Through spiking experiments with compounds other than water, it has been determined that this method has no interference from alcohols, ketones, propylene glycol, glycol esters, polyglycols and various metal oxides. However, interferences from ethylene glycol and some organic and inorganic acids have been observed.

The HydroScout system was shown to be an accurate, easy to use method for determining water in a wide variety of lubricating oils. The reagents are pre-measured, seal in glass ampules and can be disposed of in normal laboratory waste. Some cross reactivity was observed and the user must choose the program that best fits his type of oil. HydroScout can be an effective diagnostic test on a variety of oils comparing favorably with the Karl Fisher method.

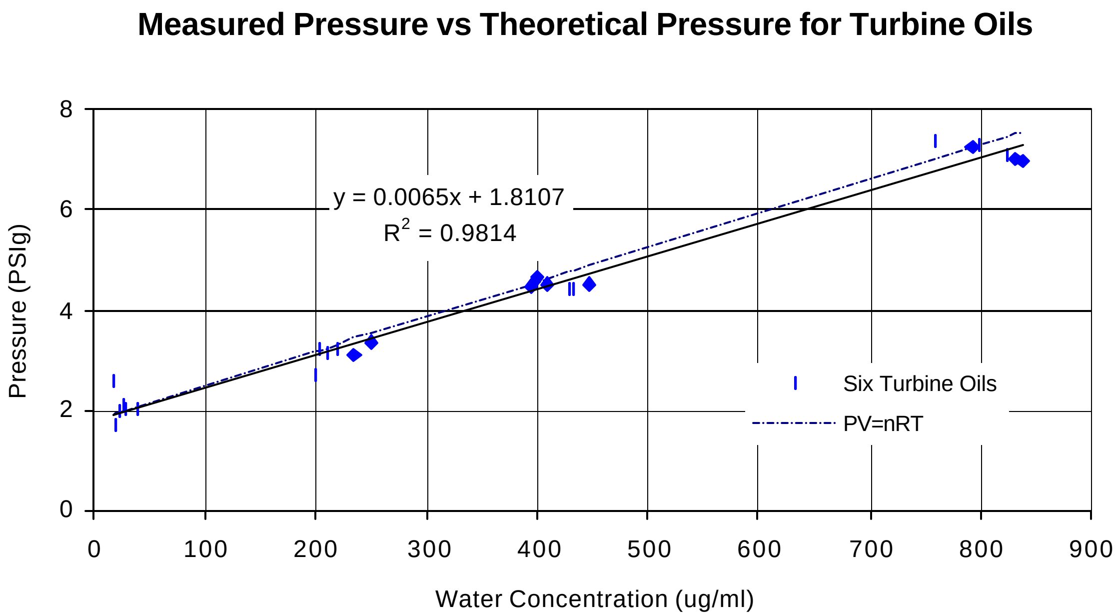

Figure 1

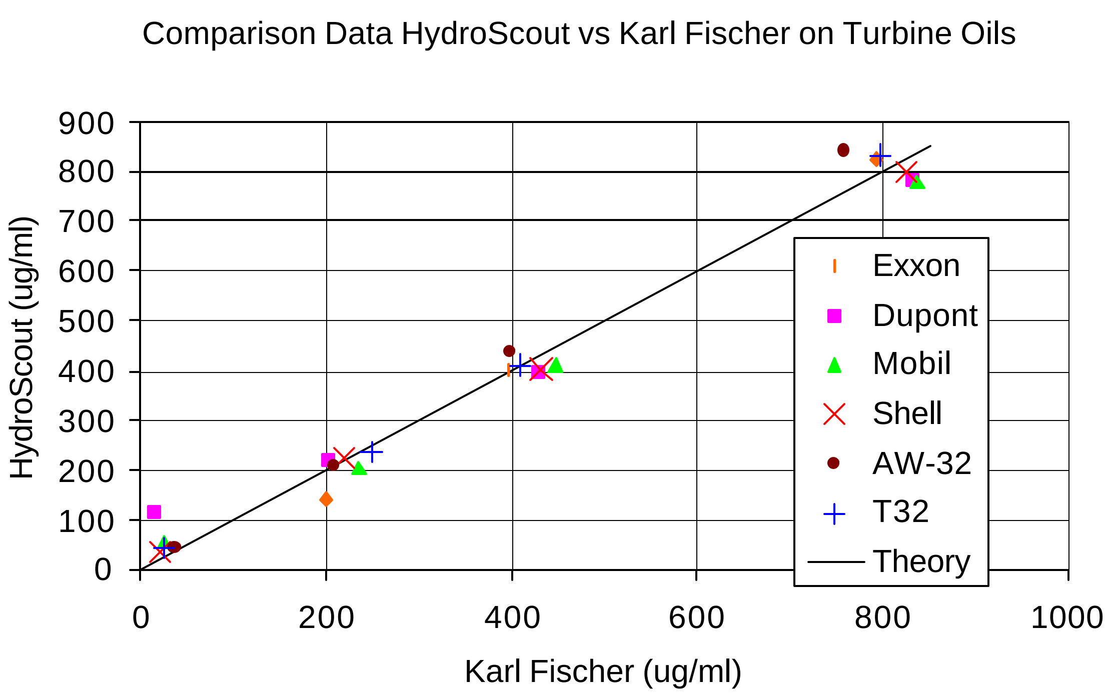

Figure 2

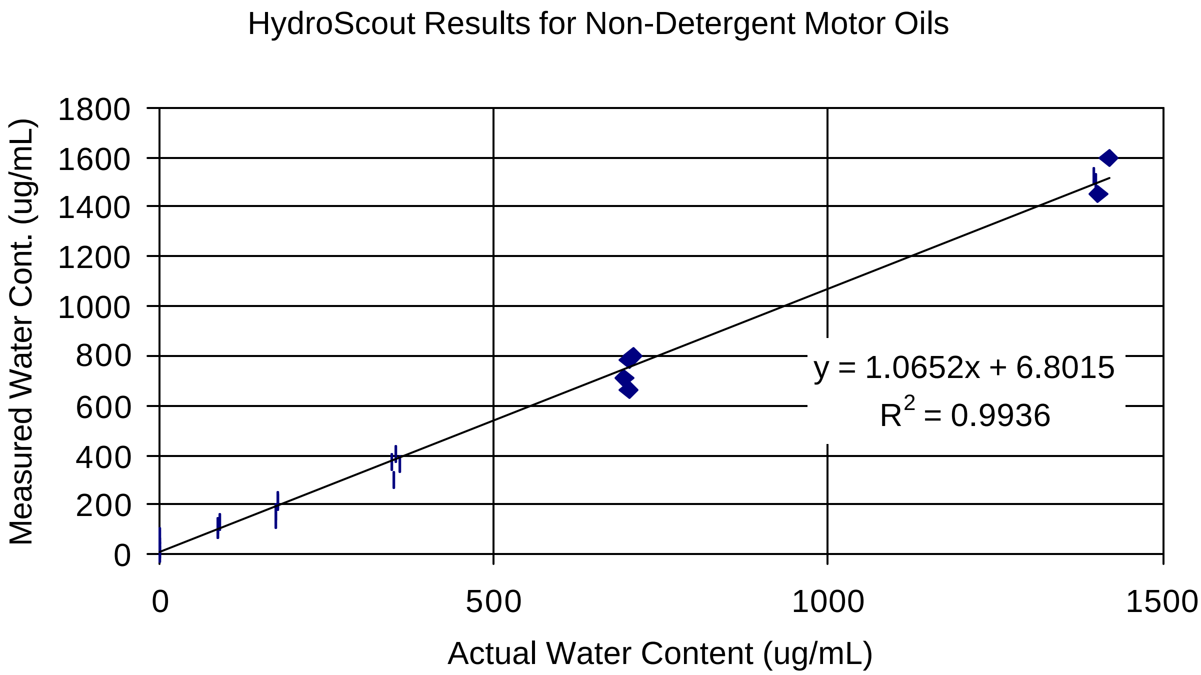

Figure 3

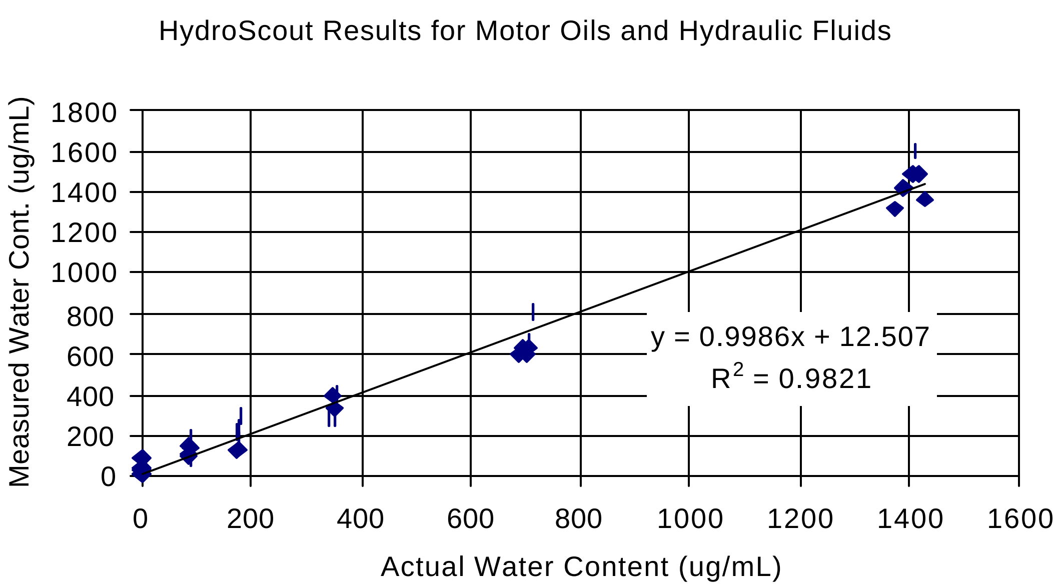

Figure 4

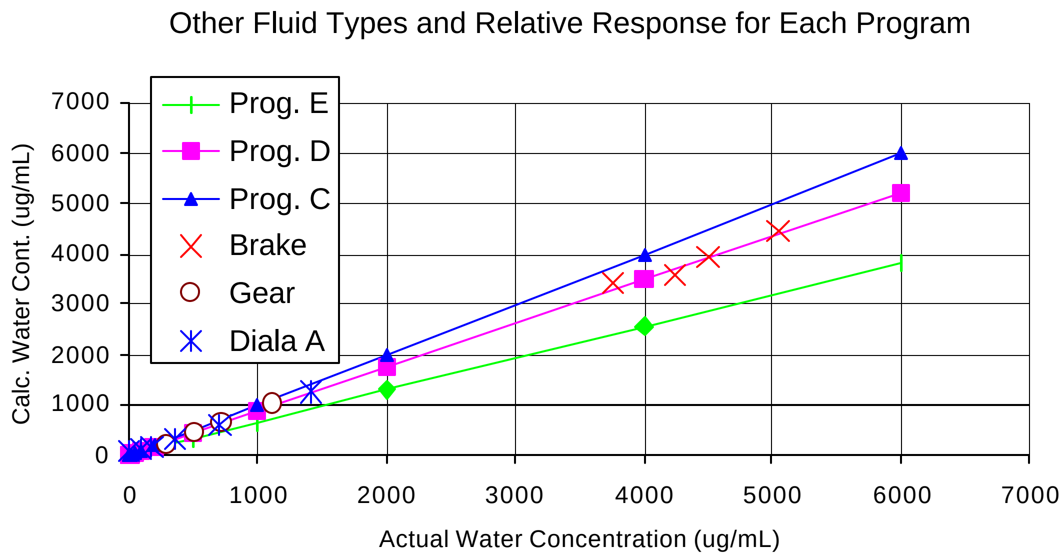
Other Fluid Types and Relative Response for Each Program
Prog. E
Prog. D
Prog. C
Brake
Gear
Diala A
7000
6000
5000
4000
3000
2000
1000
0
Calc. Water Cont. (ug/mL)
0
1000
2000
3000
4000
5000
6000
7000
Actual Water Concentration (ug/mL)

Figure 5