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WHITE PAPER

# Guide to Measuring Glycol Contamination in Oil

## Overview

Liquid cooled engines and rotating equipment use glycol based coolants because of their excellent heat transfer abilities. However glycol based coolants are not desirable at all in lubrication oil itself – Coolant ingression to the lubricant is a particularly nasty contaminant for the variety of damage it can cause.



Glycol coolants break down in the high temperature engine environment, leading to formation of glycolic acids. These acids attack nonferrous bearing surfaces and form metal salts. The acids also react with the oil anti wear and anti oxidant additives and, along with water, create sludges that plug filters and cause the oil to lose its lubricity properties, thus increasing abrasive wear. Glycol contamination in engines and transmissions is considered to be a more severe contaminant than water alone (up to 10 times more damaging). Depending on the oil temperature, the glycol coolant may break down rapidly or over time. This instability is a major challenge for determining the true glycol content in the oil at a given time, and is the major reason why field and lab tests often do not agree with each other.

Lab tests by Cummins showed that "an oil containing four percent coolant will retain only 10 percent of the glycol originally present upon heating at 200°F (93°C) for eight hours." However, other telltale signs of glycol contamination do remain in the oil.

# Methods for Measuring Glycol Contamination



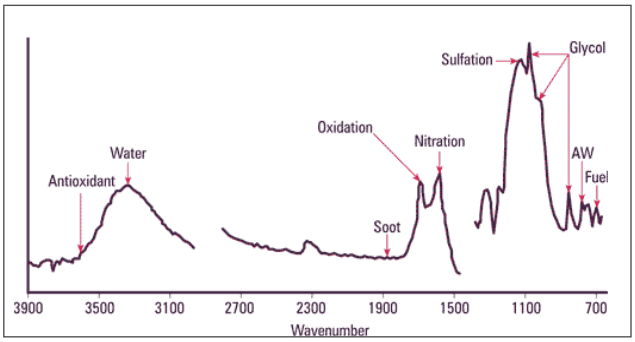
FluidScan®

## IR SPECTROSCOPY

Infrared Spectroscopy is commonly used in the lab or in the field to detect a series of molecular contaminants and lubricant chemistry degradation parameters. Infrared spectroscopy uses an Infrared radiation source, a sample holder, a detector, and a computer to study the interaction of matter and light. Molecules from various compounds vibrate in characteristic and reproducible frequencies so that a spectrum scan in the area of interest will indicate the nature and concentration of the contaminant of interest. Glycol has a strong absorbing band in the region around 3450 $cm^{-1}$ corresponding to the O-H functional group, as well as a more unique band for ethylene glycol at 1070-1030 $cm^{-1}$ corresponding to the C-O functional group. Interferences from water and oil addtives can cause errors in measurement, so a significant amount of signal processing is required to ensure reliable and consistent results.

PROS
- Sensitive to glycol
- Field tool detects liquid glycol accurately and data can be saved to monitor trends
- Easy to use
- Other parameters can be measured simultaneously (water, soot etc)

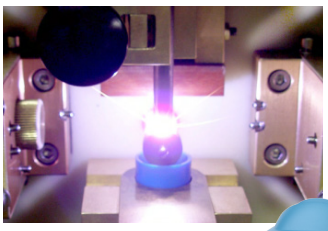
CONS
- Requires spectrum processing to determine interferences
- Liquid glycol in oil can be hard to detect
- Limit of detection not low enough for certain equipment




Typical used oil FTIR spectrum

There are test methods for laboratory grade FTIR measurement as well as for portable field testing. ASTM E2412 describes the standard practice for FTIR measurement of common engine oil degradation products and contaminants including glycol. In this test a 15 ml oil sample is pumped through an automated system to a sampling cell where the measurement is taken. The system is then flushed with heptane to prepare it for the next oil sample.

For monitoring oil chemistry in the field, ASTM D7889 uses a grating infrared spectrometer like the FluidScan which is easy to operate and does not require an experienced technician. The portable FluidScan relies on a patented flip top optical cell which only requires a few drops of oil to complete the analysis. No solvents are required to clean the flip top cell. It can be wiped down using a clean cloth or disposable wipe.



Oil sample being analyzed by Rotating Disc Electrode spectrometer.




SpectrOil 100

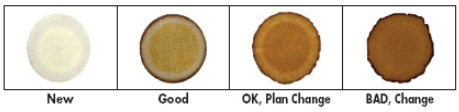
## ATOMIC EMISSION SPECTROSCOPY

Elemental analysis by Inductively Coupled Plasma (ICP) emission spectroscopy or by arc-spark Rotating Disc Electrode (RDE) emission spectroscopy has been standard practice for oil analysis testing labs for years, as detailed in ASTM D5185 and ASTM D6595. Elemental analysis is a very reliable method to verify that glycol ingression has occurred, as traces of the coolant remain in the oil, specifically the metallo-organic corrosion inhibitors that are present in high concentrations in the glycol coolant, but not native to the oil formulation. Sodium, boron, potassium and silicon are commonly added to coolant for corrosion inhibition.

For RDE, the oil sample is poured directly into a cap and a new graphite disk electrode and pencil electrode introduced to the chamber and energized. In about a minute, the oil analysis will reveal typically 24 to 30 elements, and the additive values compared to a sample of new oil. One major engine manufacturer suggests that an increase in sodium in the oil by as little as 50 ppm can mean as much as one gallon of coolant has leaked into a 10 gallon (38 L) lube oil system.

PROS
- Most reliable indicator that coolant contamination occurred
- Multielemental, 60 sec test
- Trendable
- Easy to use (RDE)

CONS
- Molecular glycol is not measured (liquid glycol in oil) – when it is most harmful
- Workshop or lab environment – not suitable for field test
- Requires knowledge of additives and coolant chemistry for diagnosis



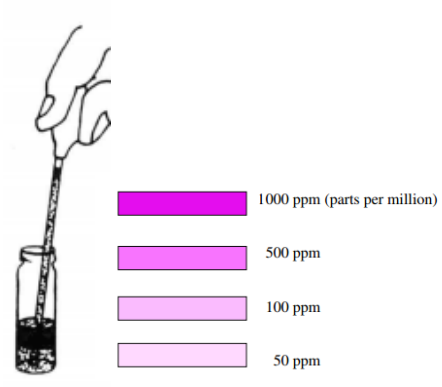

Examples of blotter tests from different oils

## BLOTTER TEST

A sheet of common blotter paper is placed on a flat surface and a dipstick is used to place a few drops of oil in the middle of the sheet and left for an hour. If the oil drop absorbs outward and there are defined "soot rings" with a yellow/brown center, that is qualitative evidence that glycol is present in the oil. It is a simple go/no go test, a common consequence of glycol contamination. A black sticky paste with a well-defined (sharp edge) periphery is cause for serious concern. Very often a soot ring develops around a yellow/brown center when glycol is present.

PROS
- Inexpensive
- Confirms presence

CONS
- Not quantitative
- Subjective
- Can't be used for trending data
- Water contamination can interfere



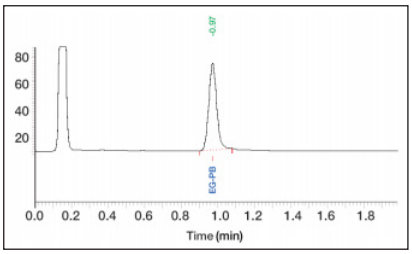

Color chart used to determine concentration of glycol in oil

## SCHIFF'S REAGENT METHOD (GLY-TEK KIT)

Perhaps the best known quick test for glycol is the Schiff's reagent method (ASTM D2982). This is a colorimetric method for detecting trace amounts of glycol in lubricating oils. In this method, a sample of oil is pipetted into a solution of hydrochloric (HCl) and iodic ($HIO_3$) acid to oxidize any glycol that may be present. The solution is then manually pipetted up a glass tube for better viewing. The reaction produces an aldehyde, which in turn reacts with the Schiff's reagent, yielding a positive color change from colorless to pink/purple – the darker the color, the more glycol present.

PROS
- Inexpensive
- Color test provides some quantitative result
- Glycol specific

CONS
- 15 minute test minimum
- Additive or refining residuals interference with some new oils
- Difficult to determine pink color with sooty/dark oils, especially transmissions oils (red to start)
- Reaction proceeds after test is read-results subjective
- Uses dangerous acids




Chromatogram of a Glycol peak in engine oil by headspace GC



Perkin Elmer Clarus 500 GC with headsampler

## GAS CHROMATOGRAPHY

The most commonly used GC procedure for glycol analysis is ASTM 4291, "Standard Test Method for Trace Ethylene Glycol in Used Engine Oil." The procedure involves first extracting the glycol from the oil using water followed by centrifugation. The aqueous extract is injected into the column, and the eluting compounds are detected by a flame ionization detector (FID) and reported out. Gas chromatography (GC) can also be used for analysis, but the ethylene glycol is difficult to detect and quantify due to its low molecular weight, low volatility and high polarity. Ethylene glycol chromatographic peak shape is often difficult to control and carryover can be a problem.

PROS
- Low limit of detection
- Glycol specific
- Easy to automate for high volume labs
- Headspace alternate methods improve accuracy

CONS
- Labor intensive preparation and unforgiving chromatography method (30 minutes per test)
- False positives due to oil additives or manufacturing byproducts
- Requires highly trained personnel
- Glycol may be already decomposed by time it reaches a lab with a GC
- Impractical for field use

## Summary

Glycol is a potent contaminant that must be monitored whenever it is employed for heat transfer duties in engines or rotating equipment. There are several direct and indirect methods for glycol detection in oil, and more than one may be required to understand if a problem is occurring.

### REFERENCES

Schaeffer Lubrication

Machinery Lubrication

Determination of Ethylene Glycol in Used Engine Oil by Headspace-Gas Chromatography , Ruppel , T, Hall G Perkin Elmer Application Note - http://www.perkinelmer.com/

Cummins Engine Fuel and Lubrication Guide - https://cumminsengines.com/



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