Analysis of 2,6 di-tertiary Butyl para Cresol Dielectric Fluid Inhibitor with a Portable Infrared Analyzer

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Introduction

The electrical transformer is an essential link in the power distribution grid. This link is necessary to step down the high voltage electricity transmission for use in businesses and homes. The performance of the transformers depends on several factors, including the condition of the dielectric fluid and the paper insulation around the copper coil windings.[1].The dielectric fluid is used for the purpose of insulation and as a cooling medium for the transformer. The most common of these liquids today is mineral oil. During the life of the transformer, the mineral oil and paper are subjected to some adverse influences that can cause it to break down. Heat, moisture and oxygen are three main factors that affect the life of the transformer. In an attempt to minimize the detrimental actions of oxygen during operation, the oxidation inhibitor 2,6 Di-tertiary Butyl *p* Cresol (DBPC) or in some cases 2,6 Di-tertiary-Butyl Phenol (DBP), is added to the mineral oil at about 0.3 weight percent (wt %). Over time, the inhibitor is used up and it becomes necessary to analyze the oil for the remaining DBPC and add an additional amount to return to the optimum value of 0.3 wt %[2]. This can be accomplished quantitatively with infrared spectroscopy. The test for the inhibitor in the mid IR range is a good choice because of the excellent absorbance of the organic chemicals in this spectral region at the exclusion of any interference from the mineral oil [3]. The absorbance of light through a medium is given by

$$A = Log\left(\frac{I_R}{I_A}\right)$$

where I_R is the intensity of the incident beam and I_A is the power of the transmitted light. See **Figure 2**

The absorbance of a solution of the DBPC inhibitor in the mineral oil adheres to Beer-Lambert Law, which states that the absorbance is a function of the concentration. Deviations from linearity can determined by obtaining absorbance values from known amounts of inhibitor in mineral oil and by creating a calibration table, see **Chart 1, Graph 1**.

Beer Lambert Law: **A = abc**

Where:

a = Wavelength dependent molar absorbtivity of a particular analyte

b = light path of the sample cell

c = analyte concentration

Since both **a** and **b** are constants for a given analyte, we can represent this value by **k**. The **k** value is the same for both the standard and the sample and, therefore, will be cancelled out. This fact allows us to represent the relationship between the inhibitor standard and the concentration in the sample by the equation:

$$\frac{A_{std}}{A_{unknown}} = \frac{C_{std}}{C_{unknown}}$$

Dielectric fluid is typically sampled in the field by a service technician and delivered to a remote laboratory for completion of the test. A study was undertaken to determine whether a portable infrared instrument could be used to analyze the dielectric fluid for inhibitor content at the field location.

Experimental Section

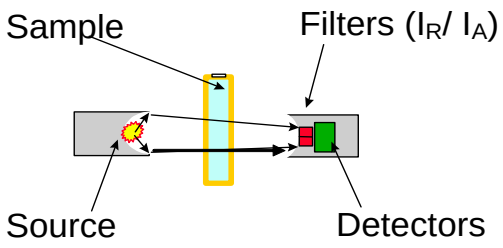
Chemicals

The mineral oil used in this experiment is uninhibited transformer oil similar to Shell Diala A. The 2,6 Di-tert–Butyl-ρ-Cresol (DBPC) also knows as 2,6 Di-tert-Butyl-4-Methylphenol used for the standards can be obtained from Fluka Chemika as Fluka number 34750. The sample cells can be cleaned with commonly available Shell VM&P Naphtha.

Instruments

The infrared analyzer spectrophotometer should be a portable, battery powered instrument that is capable of transport to remote locations void of an electrical power supply. The instrument, which is a fixed wavelength filter, should be capable of producing an absorbance reading at 3650cm^{-1}(2.7 μm) [1]. An example of such an instrument is the portable Wilks InfraCal Filtometer (see **Figure 1**). This portable instrument used for this purpose has a resolution wavelength listed at 40 cm$^{-1.}$

Figure 1 Wilks InfraCal Filtometer



$A = \log (I_R / I_A)$

Figure 2 The Measurement of IR Absorption of a transparent sample by Transmission

Procedure

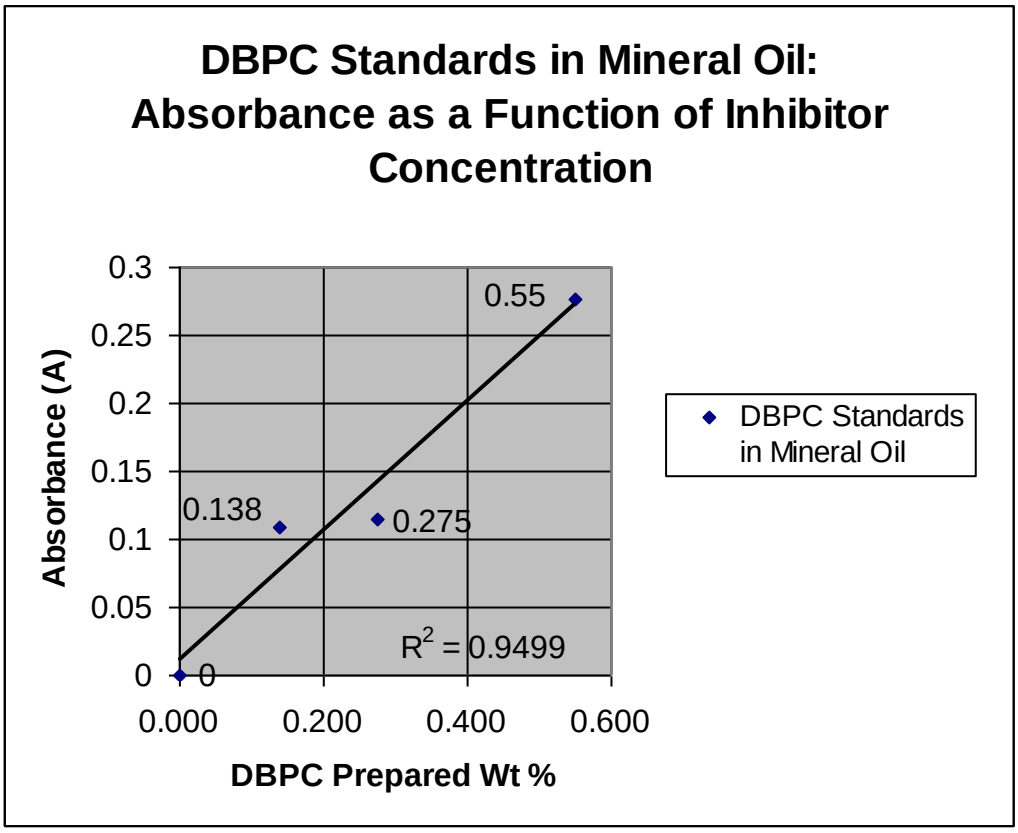
A 0.55 wt/% DBPC standard is prepared by dissolving 0.487 g of the inhibitor in 88 g uninhibited virgin oil in a 250 mL. flask, with heating, in an oven at 50°C. The flask was removed from the oven and cooled to ambient temperature before the calibration step. After the DBPC was completely dissolved, the standard was transferred to a container with a screw cap.
The infrared spectrometer was set up according to the instrument manual. This portable instrument has no optical air path, so there is no need to purge the sample compartment with an inert gas as is typically done. Calibration was initiated by first drawing up from the supply of uninhibited virgin oil used in making the DBPC standard into a 10 ml syringe and transferred into a clean sealed sample cell with a 0.5 to 1.0 mm path-length. The virgin oil was run on the instrument to determine the absorbance at 3650 cm^{-1} (2.7 µm).to establish the base line for the standards and dielectric mineral oil samples. After the virgin oil was run, the 0.55 wt % standard DPBC in virgin oil was drawn into a clean syringe and transferred two or three times into the cell to flush out the previous oil and then capped with Teflon plugs. This standard was run the same way as the uninhibited virgin oil blank and the absorbance of the DBPC inhibitor at 3650 cm^{-1}(2.7 µm) was noted for calculation.
The samples were run the same way as the standards. After the absorbance values were obtained and the absorbance of the virgin oil blank was subtracted, the concentration of the inhibitor was calculated from the ratios of the absorbance values and concentrations with the equation:

$$\frac{A_{std}}{A_{unknown}} = \frac{C_{std}}{C_{unknown}}$$

where A_{std} and $A_{unknown}$ are the absorbance values of the DBPC inhibitor standard and unknown transformer oil sample, respectively, and C_{std} and $C_{unknown}$ are concentrations of the DPBC standard and the unknown sample. The unknown concentrations were obtained by solving the above equation for $C_{unknown}$.

Results and Discussion

Several transformer dielectric fluid customer samples (used mineral oil) with DBPC inhibitor content previously analyzed at S. D. Myers, Inc with a laboratory bench top FT-IR. by ASTM method D-2668 [4] were selected for this study. These samples were run on portable infrared spectrophotometers, including one from Wilks Enterprises, Inc. and one from Laboratory A. The test protocol of the analysis specified one-point calibration standard and the accommodating the virgin oil blank baseline, as described previously. The prepared standard inhibitor concentrations used in this study were listed in **Chart 1** and plotted against the absorbance in **Graph 1** to determine adherence to the Beer Lambert Law. The results from these standards are shown below.



Graph 1 Standards Comparison

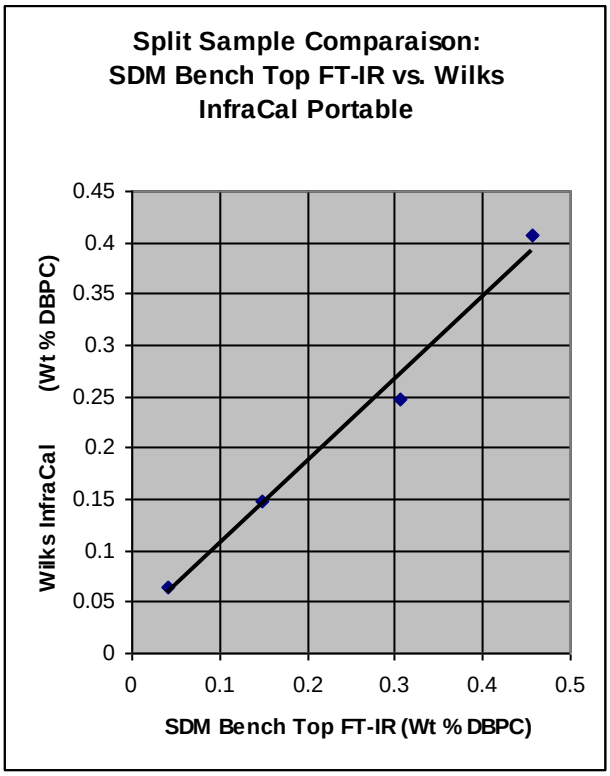
DBPC Standards & Absorbance

Sample Absorbance	DBPC Wt % Prepared
0.277	**0.55**
0.114	**0.275**
0.109	**0.138**
.0	**0**

Chart 1

The absorbance value of the inhibitor in mineral oil has been determined to be linear through and up to the prepared 0.55 wt % standard.

The samples for this study were dielectric fluids drawn from transformers in service at the time. The particular samples selected were from available samples submitted to the S. D. Myers, Inc laboratory for dielectric quality checks including inhibitor content. After the laboratory completed this test, the oil samples were tested on the commercial portable IR instruments of several companies, including the Wilks InfraCal Filtometer. The results from this **split sample** comparison, with Wilks, are shown below in both **Graph 2 and Chart 2 (last column).** The results agreed reasonably well.

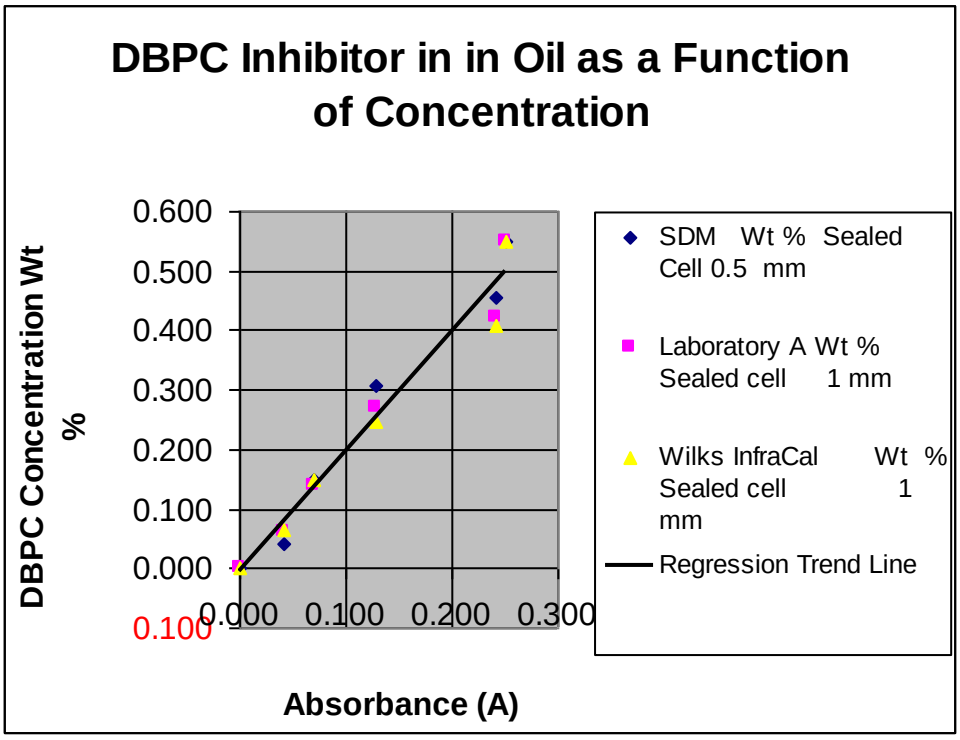


Graph 2 Split Sample comparison; SDM vs. Wilks

Sample	SDM Absorbance	SDM Wt %	Laboratory A Absorbance	Laboratory A Wt %	Wilks InfraCal Absorbance	Wilks InfraCal Wt %	Wilks-SDM Δ Wt % Split Samples)
	Sealed Cell 0.5 mm	Sealed Cell 0.5 mm	Sealed cell 1 mm	Sealed cell 1 mm	Sealed cell 1 mm	Sealed cell 1 mm	
No.112	0.0417	**0.042**	0.06	**0.06**	0.064	**0.064**	**0.022**
No.198	0.0698	**0.149**	0.066	**0.14**	0.070	**0.149**	**0.000**
No.171	0.1284	**0.307**	0.113	**0.27**	0.103	**0.247**	**-0.060**
No. 62	0.242	**0.456**	0.223	**0.42**	0.216	**0.408**	**-0.048**

Chart 2 Sample Results & Split Sample Comparison

The in house analysis for inhibitor at S. D. Myers, Inc., designated as **SDM,** was done on a bench scale FT-IR instrument and is compared to the portable instrument results. See **Chart 2**, **Graph 3**.



Graph 3

Another method for DBPC or DBP inhibitor determination is given in ASTM D 4768 [5] and is performed by Gas Chromatography (GC) using a solvent solution and an FID detector. The analysis time with sample and standard preparation is one to two days in length. The method also requires the use of flammable solvents and running five standards on the instrument before the sample is prepared and run. The proposed portable IR method requires one standard of 100 ml, which can be stored up to 6 months or longer under refrigeration. Since the portable instrument used only about one to two ml for the calibration, the standard does not have to be prepared for each analysis. The disposal of the waste material is simplified without the flammable solvent and the relatively small volume of waste oil after the test is completed.

Conclusion

The portable infrared analyzer instrument gave a good representation of the inhibitor DBPC in the mineral oil dielectric fluid used in electrical equipment over the range of the experiment of 0.0 to 0.55 weight percent. This range of inhibitor has been studied and has been determined that 0.3 % is the optimum amount for the purpose of prolonging transformer life[2]**.** Any more of this chemical in the dielectric fluid does not seem to offer any improved protection to the system and may actually cause some detrimental affect in the transformer[1]**.** The portability of the infrared analyzer instrument can be a real convenience, especially when an electrical outlet is not available for instrument connection. The analysis of the inhibitor can be carried out in less than one minute and can be performed by an operator with minimal training. This portability allows for the immediate sampling and testing of the sample without the need to draw and transport it to a remote laboratory. The portable unit would generate a relatively small volume of test oil that can be carried with the field crew until a convenient time for disposal.

Bibliography

1 *Transformer Maintenance Guide*, Transformer Maintenance Institute, S. D. Myers, Inc. 2004

2 F. C. Doble, ”The Reclamation of Insulating Oils”, ASTM STP #152 (1952)

3 P. Wilks, “IR Filtometers for Today’s Analytical Requirements”, Spectroscopy **21**(4) 43-46 (2006)

4 ASTM D-2668-02 “Standard Test Method for 2,6 di-tert-Butyl *p*-Cresol and 2,6-di-tertiary-Butyl Phenol in Electrical Insulating Oil by Infrared Absorption.

5 ASTM D-4768-03 “Analysis of 2,6-di-tertiary-Butyl Para Cresol and 2,6-di-tertiary-Butyl Phenol in insulating Liquids by Gas Chromatography.

Acknowledgement

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