

## FT-IR Spectroscopy

**Author**

Avdhut L. Maldikar, Ph.D.

Perkin Elmer, Pvt. Ltd.

Kasarvadavali, Thane (West)  
India

## Determination of Oil Content in Membranes Used in Compressed Air Sampling by Infrared Spectroscopy

**Introduction**

Compressed air sampling is very essential from an environmental point of view, wherein it is required to know all the environmental parameters such as SO<sub>x</sub> and NO<sub>x</sub>. These parameters are very important to determine the air quality. Membranes used in compressor should be oil free. Hence quantitative estimation of oil trapped in the membranes being used in air compressor is very important. FT-IR studies can be very effective in calculating the oil content in membranes to a very

low level as well. This type of work is being carried out using FT-IR in the environmental segment using such tools as the Environmental Hydrocarbons FT-IR Analysis System (<http://www.perkinelmer.com/Catalog/Product/ID/L160000S>), which includes the Spectrum Two instrument and Spectrum Touch software with an application for oil in water measurement.

This note describes the test method for the quantitative analysis of aerosol oil and liquid oil typically present in the air discharged from compressors and compressed air systems. The method is rapid, sensitive and cost effective and shows the FT-IR can be an effective tool for the monitoring of oil content. The methodology followed for the analysis by FT-IR is reported in BIS (Bureau of Indian Standard)<sup>1</sup> and we have also tested for its ruggedness, spike recovery, linearity and detection limits.

Air filtration or Air filters are used and designed into gas/air flow systems to remove particulates from the air flow. Air filters and filtration techniques are used for wide variety of purposes, some of which include:

- Improving the breathable environment of occupants.
- Elimination or minimizing fire hazards
- Removing airborne bacteria from controlled environments.

This method is applicable when an additional sampling probe is inserted to maintain isokinetic conditions from the main pipe flow. For sampling from high flow systems, isokinetic sampling may be used when wall flow is not present. Condensate may also be collected at the sampling filters. When high levels of condensates are present, collection rates increase and larger collecting containers are required.

The liquid collected consists of water, oil/water emulsion and oil. Depending upon the type of oil, separation of the oil/water emulsion may occur, allowing the water to be drained off and oil to be measured.

## Experimental

### Reagents

Carbon tetrachloride (CCl<sub>4</sub>) and 1,1,2,2, tetrachloroethylene (C<sub>2</sub>Cl<sub>4</sub>) AR Grade, used as solvent.

Precaution: Carbon tetrachloride is hazardous and should be handled with care using appropriate protective equipment. However its use is generally discouraged by the Montreal protocol due to its adverse effect on the ozone layer. Other perhalogenated solvents such as tetrachloroethylene (C<sub>2</sub>Cl<sub>4</sub>) are equally suitable provided the hydrocarbon residue is sufficiently low.

### FT-IR Parameters

FT-IR parameters was kept as shown in table 1.

**Table 1. FT-IR scanning parameters.**

Scan Range	3400 cm <sup>-1</sup> to 2500 cm <sup>-1</sup>
Resolution	4 cm <sup>-1</sup>
No. of Scans	8
Accessory	Slide holder with 50 mm path length cell



Figure 1. Spectrum Two FT-IR instrument.

### Preparation of calibration graph

**Calibration of stock solution (1 mg/mL):** 100 mg of oil was weighed in 100 mL volumetric standard flask. Weighed oil was then diluted up to the mark using carbon tetrachloride/ 1,1,2,2 tetrachloroethane as solvent. This solution was preserved as a stock solution.

**Calibration of standard solutions:** The calibration range was prepared using the stock solution. The standard solution was further diluted in accordance with Table 2. Spectra of each resulting solution were taken to get the calibration range as shown in Figure 2.

**Table 2. Calibration standards range.**

Vol. of stock sol. (mL) pipette from stock (1 mg/mL)	diluted to (mL)	Final Conc. (ppm)
0.1	50	2
0.5	50	10
1.0	50	20
2.0	50	40
2.5	50	50
3.0	50	60
3.5	50	70

Calibration graph was plotted by calculating

$$A = \log_{10}(I_0^3/I_1I_2I_3)$$

vs. concentration of oil standard in ppm, when carbon tetrachloride was used as a solvent.

$I_0$  = intensity at baseline

$I_1$  = intensity at 2960 cm<sup>-1</sup>

$I_2$  = intensity at 2925 cm<sup>-1</sup>

$I_3$  = intensity at 2860 cm<sup>-1</sup>

In the case of 1,1,2,2, tetrachloroethane calibration plot was plotted by calculating Absorbance vs concentration of oil standard in ppm. Note that the calibration graphs were valid only for the oil used and for the type of cell used.

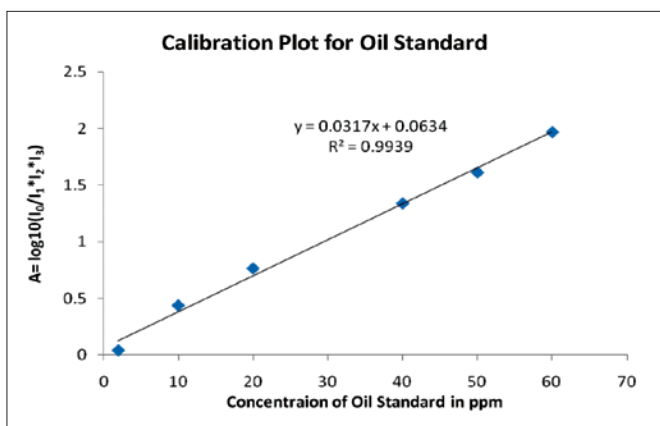


Figure 2. Calibration plot using carbon tetrachloride as solvent.

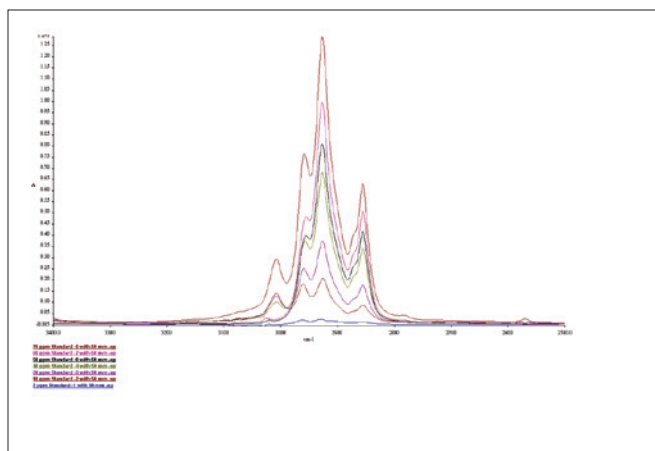


Figure 4. Standard spectra using carbon tetrachloride as solvent.

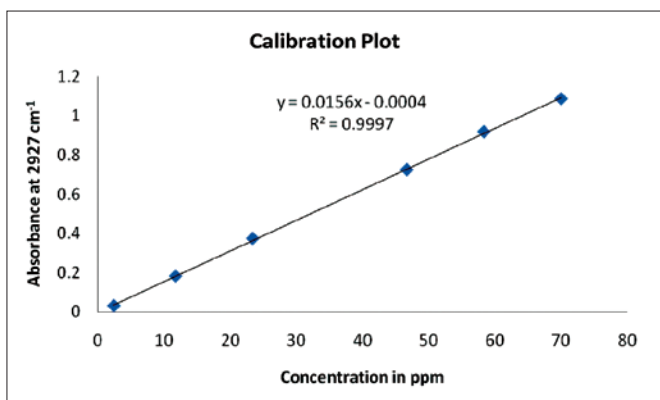


Figure 3. Calibration plot using 1,1,2,2 tetrachloroethane as solvent.

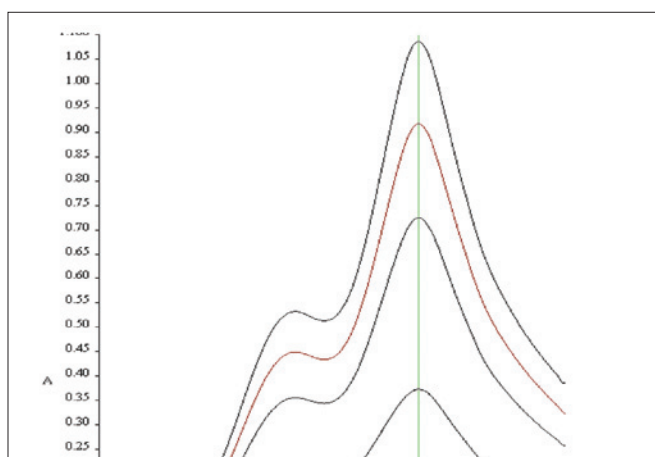


Figure 5. Standard spectra using 1,1,2,2 tetrachloroethane as solvent.

## Preparation of sample

Membrane was first accurately weighed on a balance. Further it was kept for extraction of oil in 25 mL carbon tetrachloride/1,1,2,2 tetrachloroethane solvent for 30 mins. The solution was decanted into a 25 mL standard flask and the membrane was washed thoroughly. All the washings were collected in the same 25 mL standard flask. Finally volume was made up using carbon tetra chloride/1,1,2,2 tetrachloroethane. FT-IR spectrum was recorded using the same FT-IR parameters which were used for standard calibration plot. The Absorbance (A) was calculated as per the graph and reported as unknown sample concentration. The experiment was repeated and checked for reproducibility of the method. The data demonstrates the repeatability and reproducibility of the method. Figures 4 and 5 show the spectra for unknown samples in carbon tetrachloride and 1,1,2,2 tetrachloroethane respectively.

## Results and Discussion

### Validation

#### Repeatability and reproducibility of the data

The repeatability and reproducibility test of the developed method was carried out using a 20 ppm standard solution five times and the absorbance was calculated. It was observed that data is repeatable and reproducible in both the solvents as shown in Table 3.

Table 3. Repeated analysis for 20 ppm solution.

Sr. No.	Conc. of solution (ppm)	Absorbance in CCl <sub>4</sub>	Absorbance in C <sub>2</sub> Cl <sub>4</sub>
1	20	0.77	0.32
2	20	0.78	0.35
3	20	0.78	0.36
4	20	0.78	0.31
5	20	0.78	0.31

## Working Range

The working range for the method was found to be 1 ppm to 70 ppm provided that a 50 mm pathlength cell has been employed.

## Calculations

$$X = (A * V_s) / (C * q * H * 3600)$$

Where;

A: absorbance

V<sub>s</sub>: volume in mL of solvent used for extraction

C: calibration coefficient

q: flow rate in Ltr/sec

H: duration in hrs

The above formula was mentioned in ISO 8573-2 method.

## Conclusions

The developed method is sensitive and cost effective and shows that FT-IR can be an effective tool for the monitoring of oil content in membranes. It does not require complex sample preparation. Spiking studies can be studied using the sample solutions and with three levels of C1, C2 and C3. The instrument, Spectrum Two gives us the flexibility to run the samples in liquid phase with 50 mm path length cells. This application is useful in environmental segment wherein we need to monitor the quality of air compressors in terms of oil being trapped. This technique is really helpful for air compressor manufacturers to test the trapped oil during air sampling.

## References

1. Compressed Air for General Use, BIS 1999.
2. Compressed Air – Part 2 Test Methods for Aerosol Oil Content, ISO-8573-2, 2007.
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