Anton Paar

SVM™ 3001 with Abbemat for Transformer Oils

Relevant for: Petroleum Industry
Production of base oils, formulation of transformer and electrical insulation oils. Manufacturers of transformers and power plants.

Determine the carbon type composition according to ASTM D2140 and viscosity parameters according to ASTM D3487 and IEC 60296.

1 Introduction

Transformer oils have two important functions: to be an electric insulator and to act as a coolant for the transformer. Effective cooling will increase the lifetime of the transformer, so the cooling liquid must be able to transfer as much heat as possible.

Viscosity and density at different temperatures from -40 °C to 100 °C are essential for the characterization of insulation oils (e.g. measurements according to IEC 60296, ASTM D3487) and can be measured with the SVM™ 3001 fast and accurately.

Additionally, the carbon type composition is an important parameter for transformer oils. Critical product performance properties correlate with the carbon type composition. The Standard Practice ASTM D2140 (VGC-r_i method; viscosity gravity constant - refractivity intercept) "Calculating Carbon-Type Composition of Insulating Oils of Petroleum Origin" is used to determine the carbon-type composition of insulating oils.

To calculate the carbon type composition of an oil, following basic parameters are required:

- kinematic viscosity at 37.78 °C (100 °F) (obtained from SVM™ 3001)
- refractive index at 20 °C (obtained from the refractometer)
- density at 20 °C (calculated from API functions by the SVM™ software)
- specific gravity at 15.56 °C (60 °F; calculated by API functions of the SVM™ software)

To determine the carbon type distribution according to ASTM D2140, further the viscosity-gravity-constant (VGC according to ASTM D2501) and the refractivity intercept are required. These parameters are calculated by the SVM™ 3001 software.

This report describes specifically how to test oils with the SVM™ 3001 (according to ASTM D7042, D4052 and D2501) in combination with an Abbemat refractometer to get the carbon type composition according to ASTM D2140.

2 Instrumentation

2.1 SVM™ 3001

For the viscosity and density measurement a manually filled SVM™ 3001 Stabinger Viscometer™ is used.

Fig. 1 SVM™ 3001 with Abbemat 550

2.2 Instrument Software

The instrument software provides all required calculations by itself without any need for an external PC:

- the VGC from the specific gravity (SG) at 15.56 °C (60 °F) and from the kinematic viscosity at 37,78 °C (100 °F)
- the refractivity intercept from the refractive index at 20 °C (68 °F) and from the density at 20 °C (68 °F)

and determines from these parameters the carbon type composition.

If the oil has a sulfur content of 0.8 % or higher, a sulfur correction must be applied to improve the accuracy.

To obtain the kinematic viscosity of insulation oils according to ASTM D3487, IEC 60296 and IEC 61868, SVM™ 3001 provides temperature scan measurement modes. Scan results are available as graphic evaluations, too.

2.3 Abbemat Refractometer

For the RI measurement the Abbemat 500 is used. Connected via CAN interface, it is a module controlled by the SVM™ 3001 as master instrument.

Instead of Abbemat 500 any other model of the Abbemat Performance and Performance Plus line (300/350 or 550) respectively from the Heavy Duty line (450, 650) of Anton Paar refractometers can be used.

2.4 Installation

To install your SVM™ 3001 with Abbemat Refractometer, a connection kit is required. For parts and installation refer to the respective instruction and reference manual(s).

The measuring cells of both instruments can be filled comfortably via hose connections and a flow-through cell for the Abbemat. If only less sample is available, SVM™ 3001 and Abbemat can also be filled separately.

Find all available options in the SVM™ Product Description List (Doc. No. D89IE001).

3 Measurement

3.1 Sample and Instrument Preparation

No special sample preparation is required.

If the sample is not freshly drawn from a production line or else, homogenizing the test specimen might improve the measurement repeatability. Proceed as follows: Fill approx. 100 mL of sample into a glass beaker, cover it with a laboratory film to avoid contamination and stir the sample on a magnetic stirrer at low speed for approx. 5 min.

Ensure that the measuring cells respectively all hose connections are leak tight, clean and dry.

3.2 Settings

3.2.1 Carbon Type Composition

Method: SVM + Abbemat

SVM™ 3001:

According to ASTM D7042, following settings are predefined by default

- Measuring temperature: 37.78 °C
- Precision class "Precise"
- RDV limit 0.10 %
- RDD limit 0.0002 g/cm³
- 5 determinations
- Automatic prewetting: yes
- Sulfur correction: activated (if applicable, enter here the sulfur content of the oil)

Drying time:

- built-in air pump: 150 s
- compressed air at 2 bar: 60 s

Abbemat refractometer:

The method SVM + Abbemat includes the following settings for the refractometer:

- Temperature: 20 °C
- Measurement accuracy "Most Precise"
- Hold time: 1 s
- Timeout: 200 s
- Wavelength: 589.3 nm (fixed parameter)

3.2.2 Low Temperature Viscosity Measurement

These tests can easily performed with a temperature scan. Select the method " Standard" and change the settings as listed below:

- Measurement mode: Temperature Table Scan
- Measuring temperatures: as required
- Precision class "Precise"
- Equilibration time: 120 s
- Automatic prewetting: yes

Drying time:

- built-in air pump without drying cartridge: 120 s
  Not recommended for drying at cell temperatures below the dew point.
- built-in air pump and drying cartridge: 120 s
  For occasional drying below the dew point.
- compressed air at 0.3 bar with air preparation set dew point -40 °C: 120 s
  For drying below the dew point respectively at low temperatures.

Counter cooling and air preparation equipment:

Measurements down to -20 °C do not require external counter cooling. For better cooling performance, liquid counter cooling can be optionally connected.
For measurements below -20 °C external liquid counter cooling is required.

If the last temperature of a table scan is higher than the dew point, air drying equipment is not explicitly required. For occasional drying below the dew point a drying cartridge is sufficient. For permanent drying below the dew point or at low temperatures the air preparation set dew point -40 °C is required.

For low temperature setup and options consult the SVM™ X001 Reference Manual and - if applicable - the instruction manual of your counter cooling device.

3.3 Calibration

Before measuring the samples, it is advisable to check both instruments for correct measurement.

Perform a calibration. If required, apply a calibration correction to improve the reproducibility on the SVM™ 3001. Use one or more standard(s) in the viscosity range of your oil sample(s). This can be a certified standard or a proprietary standard with kinematic viscosity values. In any case you need reliable kinematic viscosity values at the measuring temperatures.

To perform the calibration, refer to the SVM™ 3001 Manual.
Alternatively, a calibration at -20 °C respectively -40 °C can be ordered from works (100444 Special Calibration SVM™ (-20°C) full range (5 points); 100445 Special Calibration SVM™ (-40°C) full range (5 points)).

Check also the measurement accuracy of the Abbemat by performing at least a water check. For checks and adjustment of the Abbemat refer to the instruction manual of this instrument.

3.4 Samples

- Nytro 4000X - Severely Hydrotreated Insulating Oil
- T110 - Severely Hydrotreated Base Oil
- Nyflex 3150 - Severely Hydrotreated Process Oil
- Nypar 315 - Severely hydrotreated Process Oil
- Nytro 10XN - Severely Hydrotreated Insulating Oil

For this report, the samples were kindly provided by Nynas AB, Sweden.

3.5 Filling

Syringes:

Single-use syringes are sufficient. Never use syringes with rubber sealings, as the rubber is chemically not resistant and these syringes tend to suck bubbles. To have enough sample volume for perfect prewetting and for refills, use a 10 mL syringe.

Sample volume:

- Flow-through filling: min. 3.5 mL
  typical: 6 mL

The sample volume can vary.

Sample Throughput:

- approx. 8 samples/hour

3.6 Cleaning

3.6.1 Solvents

Solvent quality

In any case the used solvent needs to dry up completely at the measuring temperature. If using a single solvent, the solvent quality shall be "chemically pure“or "for synthesis“. If using two solvents, only the second solvent needs to meet this quality.

Cleaner's naphtha (petroleum benzine)

So-called cleaner's naphtha (petroleum benzine, a dearomatized hydrocarbon solvent, blend of mainly C7, C8, C9 n-alkanes) with a boiling range of 100 °C to 140 °C is the best choice for most oils. This universal solvent can be used over the entire temperature range of the SVM™ 3001.

Aromatic solvents

Some oil samples may require toluene or xylene, as they are not (completely) soluble in petroleum benzine. In this case petroleum benzine is recommended as second solvent for perfect drying of the cells. If petroleum benzine is not available in your country, toluene/xylene as first solvent and n-hexane/n-heptane or a similar hydrocarbon solvent (mixture) as second solvent can be used.

Ethanol, Acetone

Ethanol or acetone as second solvent are not recommended for petroleum based oils, as subsequently the surface wetting behavior of oils is worse compared to using a hydrocarbon solvent.

3.6.2 Effective Cleaning Procedure

For details refer to the SVM™ Manual.

- Remove the sample from the cells (push through or suck back) using a syringe.
- Fill approx. 4 mL of solvent using a syringe and leave the syringe connected.
- Move the plunger of the syringe forth and back when the motor is at filling speed; this way you improve the cleaning performance both in the density oscillator and in the viscosity cell.
- Before filling solvent for a new cleaning cycle, remove the sample-solvent-mixture from the cells an. Blow air through the cells for some seconds for better removing the liquid.
- Ensure to perform a sufficient number of cleaning cycles as the sample is rather highly viscous compared to the solvent.
- Perform a final flush with fresh solvent to remove any residues.
- Allow a sufficiently long drying time to be sure that the solvent can dry up completely.

Solvent consumption

- typically 12 mL
  (can vary depending on oil type and viscosity)

4 Results

For this report, the measurement and calculation results obtained from SVM™ 3001 and Abbemat 500 and the reference values on the respective data sheets (PDS, CoA) are compared.

Carbon Type Composition

Table 1: ASTM D2140 (VGC-r_i) Carbon Distribution
(mean of 4 measurements)

Sample	C_A [%]	C_N [%]	C_P [%]
T110	14.50	33.25	52.20
Nypar 315	1.38	30.00	68.60
Nyflex 3150	9.33	30.05	60.65
Nytro 4000X	2.63	44.98	52.40

Table 2: Carbon Distribution - Deviation to typical sample values *
(dev. in percentage points)

Sample	C_A		C_N		C_P	
	typ.	dev.	typ.	dev.	typ.	dev.
T110	11.7	2.80	38.2	-4.95	50.1	2.10
Nypar 315	1	0.38	34	-4.00	65	3.60
Nyflex 3150	7	2.33	33	-2.95	60	0.65
Nytro 4000X	4	-1.38	45	-0.02	51	1.40

Refractive Index

Table 3: Refractive Index and deviation to typical values at 20 °C

Sample	RI meas [nD]	RI typ. [nD]	dev [nD]
T110	1.503635	1.502	0.0016
Nypar 315	1.468229	1.468	0.0002
Nyflex 3150	1.495011	1.494	0.0010
Nytro 4000X	1.474635	n.a.	n.a.

ASTM D2501 Viscosity-Gravity Constant:

Table 4: Viscosity-Gravity Constant

Sample	meas.	typical	dev
T110	0.85587	n.a.	
Nypar 315	0.80144	0.804	0.00256
Nyflex 3150	0.83008	0.829	0.00108
Nytro 4000X	0.83852	n.a.	

Kinematic Viscosity:

The tables contain viscosity results of two insulating oils meeting the requirements according to the standards ASTM D3487 respectively IED60296 (here stated: Ed. 3). Both oils are specified to -30 °C, where Nytro 10XN has a more strict low temperature specification for kinematic viscosity given in its data sheet.

Table 5: Kinematic viscosity according to specifications in ASTM D3487

Sample	Temp. [°C]	meas. kin. vis. [mm²/s]	specified max. [mm²/s]	meets range value
Nytro 4000X	100	2,3800	3	OK
	40	9,0995	12	OK
	0	57,048	76	OK
Nytro 10XN	100	2.0684	3	OK
	40	7.5818	12	OK
	0	46.275	76	OK

Table 6: Kinematic viscosity of two insulating oils according to specifications in IEC60296

Sample	Temp. [°C]	meas. kin. vis. [mm²/s]	specified max. [mm²/s]	meets range value
Nytro 4000X	0	57.255		
	-20	272.45		
	-30	**769.28**	**1800**	**OK**
	-40	2704.7		
Nytro 10XN	0	46.462		
	-20	229.60		
	-30	**687.31**	**800 ***	**OK**
	-40	2669.4		

* Nytro 10XN max. 800 mm²/s at -30 °C according to its product data sheet.

To improve the reproducibility, a calibration correction with a suitable oil in your viscosity range may be helpful.

5 Literature

- ASTM D2140: Standard Practice for Calculating Carbon-Type Composition of Insulating Oils of Petroleum Origin
- ASTM D2501: Standard Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils
- ASTM D7042: Standard Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)
- ASTM D3487: Standard Specification for Mineral Insulating Oil Used in Electrical Apparatus
- IEC 60296: Fluids for electrotechnical applications - unused mineral insulating oils for transformers and switchgear
- EN ISO 3104: Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity
- ASTM D445: Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
- IEC 61868: Mineral insulating oils - Determination of kinematic viscosity at very low temperatures
- GOST 33-82: Nefteprodukty. Metod opredeleniia kinematicheskoi i raschet dinamicheskoi viazkosti (Petroleum products. Method for determination of kinematic viscosity and calculation of dynamic viscosity)
- Anton Paar Application Report Carbon Type Distribution of Petroleum Oils with SVM™ 4001 and Abbemat Doc. No. D89IA012EN.

6 Conclusion

The assembly of SVM™ 3001 with Abbemat is perfectly suitable for determining the carbon type composition and the low temperature viscosity of insulating oils, provided that all requirements according to 2, "Instrumentation" and 3, "Measurement" are fulfilled.

Contact Anton Paar GmbH:

Tel: +43 316 257-0
Fax: +43 316 257 257
support-visco@anton-paar.com
www.anton-paar.com

Appendix A – Transformer Oils

Most transformer oils are mineral based, they are normally obtained by fractional distillation and following hydrotreatment. There are two main groups: Naphthenic and paraffinic insulation oils.

Further it can be distinguished between inhibited and not inhibited oils. Inhibited oils contain additives, which improve the oil properties, such as pour point depressants, corrosion inhibitors and more.

Naphthenic oils have an excellent low temperature behavior (e.g. low viscosity) while paraffinic types have a higher pour point due to their waxy content. So paraffinic oils need a pour point depressant if they are used at low temperature.

Meanwhile there are also other types of insulating oils available, e.g. synthetic esters or natural esters, but they are not part of this application report.

A.1 Carbon Type Composition

Carbon-type analysis expresses the average amount of carbon atoms which occur in aromatic, naphtenic and paraffinic structures, reporting

- the percent of the carbon atoms in aromatic ring structures ($\%C_A$),
- the percent in naphthenic ring structures ($\%C_N$) and
- the percent in paraffinic chains ($\%C_P$).

There are several physical property correlations for carbon type analysis.

Besides the VGC-ri method (viscosity gravity constant – refractivity intercept) according to ASTM D2140, a further empirical procedure exists, which comes into use mainly for process oils, the n-d-M method (refractive index – density – mean relative molecular mass), standardized as ASTM D3238. See the AP Application Report "Carbon Type Distribution of Petroleum Oils with SVM™ 4001 and Abbemat", Doc. No. D89IA012EN, available on the AP Extranet.

A.2 ASTM D2140 (VGC-r_i)

“Standard Practice for Calculating Carbon-Type Composition of Insulating Oils of Petroleum Origin”. It is intended for use with fresh oils, inhibited or not inhibited and applies to oils with average molecular weights from 200 to above 600, and 0 to 50 aromatic carbon atoms.

Viscosity, density, relative density (specific gravity) and refractive index are the only experimental data required for use of this method. From these measured properties the viscosity-gravity constant (VGC) and refractivity intercept (r_i) are obtained by calculation.

A.2.1 Viscosity Gravity Constant (VGC)

The viscosity-gravity constant is a useful function for the approximate characterization of the viscous fractions of petroleum. It is relatively insensitive to molecular weight. Values of VGC near 0.8 indicate samples of paraffinic character, values close to 1.0 indicate a preponderance of aromatic structures.

Mineral oils are classified as follows:

Mineral oil	VGC
Paraffinic mineral oil:	0.790 – 0.819
Slightly naphthenic mineral oil:	0.820 – 0.849
Naphtenic mineral oil:	0.850 – 0.899
Slightly aromatic mineral oil:	0.900 – 0.939
Aromatic mineral oil:	0.940 – 0.999
Highly aromatic mineral oil:	1.000 – 1.049

The VGC is calculated from specific gravity and kinematic viscosity.

A.2.2 Refractivity Intercept (r_i)

From the measured density and refractive index the refractivity intercept is calculated according to the formulae in ASTM D2140 (respectively according to ASTM D2159). The calculated values of VGC and r_i are used with the nomogram below, to correlate those parameters with carbon-type composition.

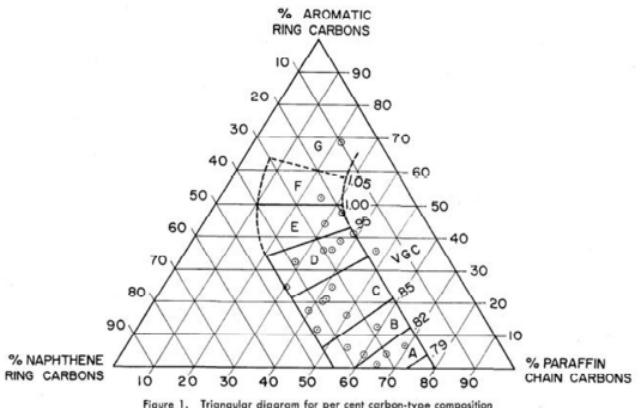

Fig. 2 Carbon type composition

A.2.3 "SVM™ + Abbemat" Method

This fast and accurate method serves mainly the basic parameters and the analysis result for carbon type composition according to ASTM D2140.

If additional useful parameters for oil characterization are required, SVM™ 3001 serves a further method "Viscosity Index + Abbemat"

Besides measurement of the required basic parameters for calculation, this method offers e.g.:

- Kinematic viscosity at 40 °C and 100 °C (extrapolated according to ASTM D341)
- Viscosity Index (according to ASTM D2270)
- Carbon type distribution and ring content according to ASTM D3238
- Mean molecular mass following ASTM D2502
- Density 20 °C
- API Spec. Gravity 15.56 °C (60 °F)

The "Viscosity Index + Abbemat" method is a temperature scan method.

For further tests on viscosity behavior of an oil, also a time scan mode is available.

A.3 Viscosity

Transformer oils act as coolant and insulation oil. Besides other properties, viscosity is a main parameter influencing the heat transfer and therefore the temperature rise within the transformer. The lower the viscosity, the easier the oil can circulate which improves heat transfer. Depending on the cooling system of the transformer, higher viscosity at low temperatures is a critical factor for the cold start of a transformer, especially for models with natural oil circulation. No or bad circulation can lead to overheating at the hot spots. and it has negative influence on the speed of moving parts, such as in pumps or power circuit breakers.

Generally, oils with a higher content of naphthenic molecules will decrease their viscosity faster than oils with a high content of paraffinic structures. Napthtenic oils have also a better heat transfer coefficient than paraffinic oils with similar viscosity at 40 °C. Additionally, naphthenic oils provide excellent low temperature behavior as they have a significantly lower pour point compared to paraffinic types. This improves the pumpability of the cold oil and allows starting a transformer at the lowest possible temperature.

Producers of such oils state in their product data sheets besides other parameters also kinematic viscosity at different temperatures (which must meet specifications according to several standards) and often the Viscosity Index as an indicator for the viscosity-temperature behavior of the oil.

Viscosity limits are defined in ASTM D3487 and IEC 60296 (here stated: Ed. 3):

Table 7: Property requirements (excerpt: viscosity) according to ASTMD3487

Temp [°C]	**Kinematic viscosity, max. [mm²/s; CSt]**	
	Type I	Type II
100	3.0	3.0
40	12.0	12.0
0	76.0	76.0

Table 8: IEC 60296: Maximum viscosity and pour point of transformer oil at lowest cold start energizing temperature (LCSET)

LCSET [°C]	**Max. viscosity [mm²/s]**	**Max. pour point [°C]**
0	1800	-10
-20	1800	-30
-30 *	**1800**	**-40**
-40 **	2500	-50

* Standard LCSET for low temperature transformer oils. Can be modified according to the requirements due to the climate in the respective country. The pour point always should be at least 10 K below LCSET.

** Standard LCSET for low temperature switchgear oil. The kinematic viscosity should not exceed 400 mm²/s at the LCSET.

Appendix B – Measuring Principles

B.1 Traditional Viscosity Determination

Traditionally kinematic viscosity is determined using glass capillary viscometers (see Fig. 3; either with manually operated or with automated systems) according to the standards ASTM D445 or ISO 3104.

The kinematic viscosity value is calculated by multiplying the efflux times with the capillary constant and - if required - by applying correction factors.

Fig. 3
Glass capillary viscometer (Ubbelohde type)

B.2 Viscosity Determination with SVM™ 3001

The Stabinger method (ASTM D7042) determines the dynamic viscosity, kinematic viscosity and density. In Fig. 4 the SVM™ principle (a modified Couette principle) can be seen: A sample-filled tube rotates at controlled speed in a copper housing. A lightweight rotor with a magnet inside floats in the sample fluid free of friction. It is driven by the fluid's shear forces and centered by centrifugal forces due to its low density. The rotor magnet induces eddy currents in the copper housing which retard the rotor. So an equilibrium speed establishes. It is a measure for the dynamic viscosity.
An integrated oscillating U-tube cell simultaneously measures the fluid's density for automatic calculation of the kinematic viscosity.

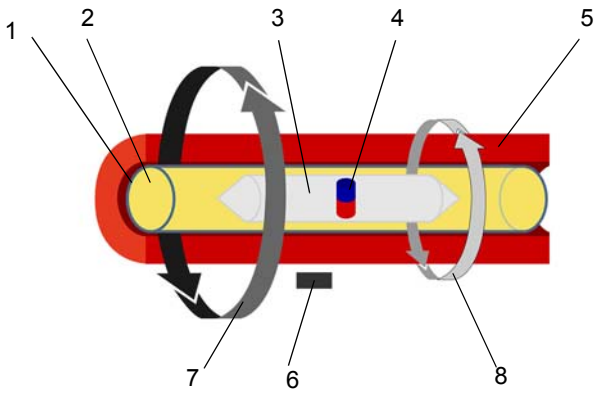

Fig. 4 Viscosity Measuring principle SVM™ 3001

1 Measuring tube
2 Sample liquid
3 Rotor
4 Magnet
5 Cell block (copper)
6 Hall-effect sensor
7 Controlled tube speed
8 Rotor speed

B.3 Refractive Index Determination with Anton Paar Abbemat

Anton Paar refractometers use reflected light to measure the refractive index. The sample on top of the measuring prism is irradiated from different angles by a light source. At the interface between sample and prism, the incident beam is either refracted into the sample or reflected back into the prism. The reflected beam is detected by a sensor array. From this the critical angle of total reflection is calculated and used to determine the refractive index (RI) of the sample. The refractive index is measured relative to air at 1013 hPa and 50 % relative humidity.

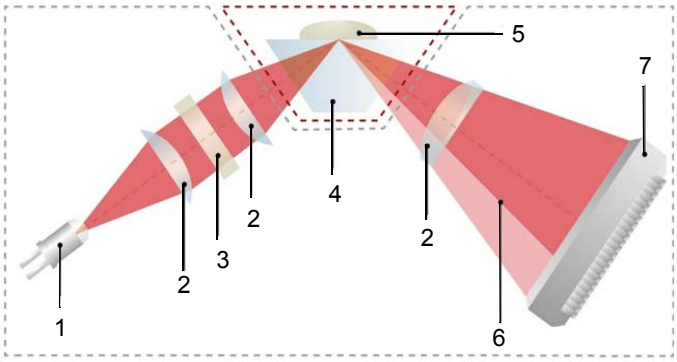

Fig. 5 Abbemat Measuring Principle

1 Light source
2 Lens
3 Interference filter
4 Prism
5 Sample liquid
6 Dark/bright borderline
7 CCD Sensor

B.4 Determination of Carbon Type Distribution by IR Spectroscopy

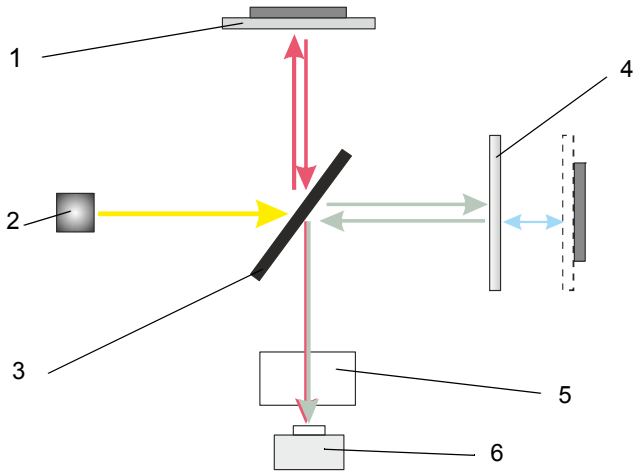

Fig. 6 FTIR functional principle (schematic)

1 Fixed mirror
2 IR light source
3 Beam-splitter (semipermeable mirror)
4 Moving mirror
5 Sample compartment
6 Detector

IR spectroscopy is a tool in organic structure determination and verification. Chemical bonds in different environments will absorb varying intensities and at varying frequencies. The frequencies at which there are absorptions of IR radiation (“peaks” or “signals”) can be correlated directly to bonds within the compound in question.

An FTIR (Fourier Transform Infra Red) spectrometer is used to apply the Brandes method (IEC 60590) in an IR spectroscopy procedure to determine carbon distribution. This enables users to investigate mineral oils with an average molecular weight between 290 and 500 and carbon in paraffinic bonds between 40 % and 70 %, as long as the carbon in aromatic bonds is below 25 %. Since chemical bonds absorb infrared energy at certain frequencies (and/or wavelengths), the basic structure of bonds can be determined by means of the IR absorption’s spectral layers.

Correlations have been found between certain absorption patterns in the infrared and the concentrations of aromatic and paraffinic carbons. The absorptions at 1610 cm^{-1} and at 725 cm^{-1} are directly related to the aromatic and paraffinic carbon concentrations, respectively.

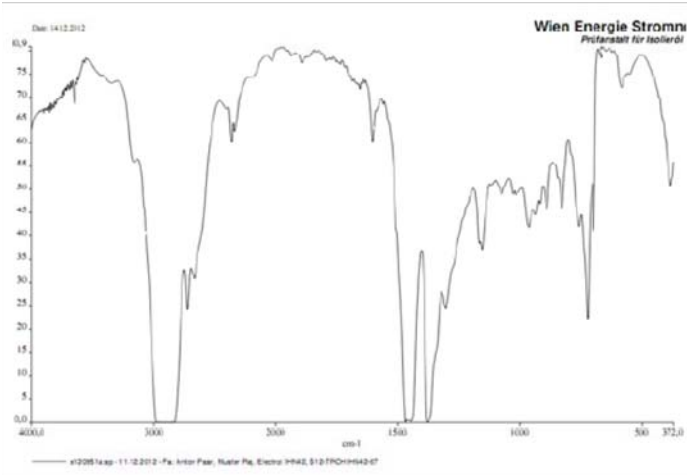

Fig. 7 FTIR Spectrogram (example)