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APPENDICES

APPENDIX A
TEST METHOD

A1. Kinematic viscosity (ASTM D-445) [11]

Kinematic viscosity is the calculated measurement of the resistive flow of fluid, based on the time in seconds required for a fixed amount of fluid to flow through a calibrated capillary tube. The time is measured for a fixed volume of sample, contained in a glass viscometer to flow through a calibrated capillary at a closely controlled temperature.

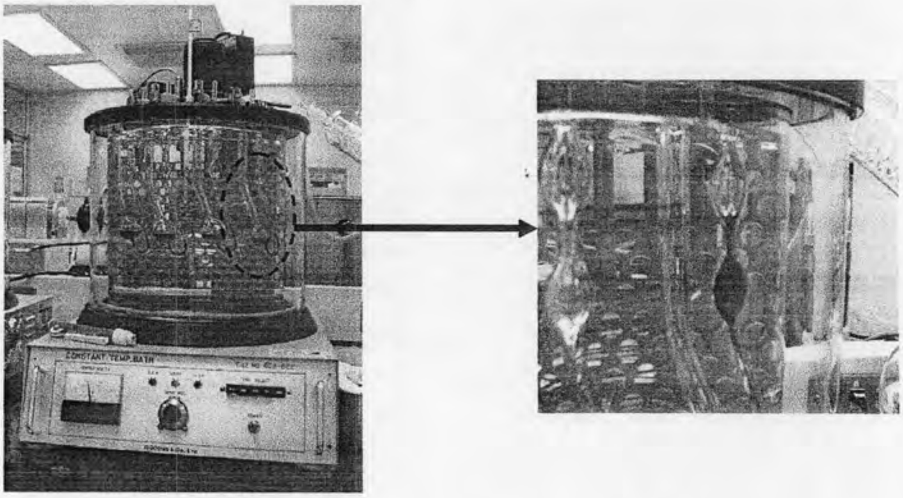


Figure A1 Kinematic viscosity instrument

- Purpose

Kinematic viscosity are required for use in all bearing design calculations and used commercially throughout the world and applied in oil blending procedures.

- Definition

Kinematic viscosity is the resistance to flow of a fluid under gravity.

- Scope of test

1.1. This test method specified a procedure for determination of kinematic viscosity (ν) of liquid petroleum, transparent liquid, by measuring the time of a volume of liquid to flow under gravity through a calibrated glass capillary viscometer.

1.2. The range of kinematic viscosities covered by this test method is from 0.2 to 300,000 mm^2/s at all temperature.

- Test method

General Procedure for kinematic Viscosity

1. Adjust and maintain the viscometer bath at the required test temperature. The temperature of the bath does not vary by more than +/- 0.05 °C of the selected temperature.
2. Thermometer shall be held in an upright position under the same condition of immersion when calibrated.
3. Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity.

Procedure for Transparent Liquids

1. Charge the viscometer with 7 ml of sample. If the sample contains solid particle, filtrate through Millipore filter (5 um or less than) before charging.
2. Allow the charged viscometer to retain in the bath long enough to reach the test temperature. Thirty minutes should be sufficient.
3. After the sample has reached temperature equilibrium, use suction to adjust the head level of test sample to a position in the capillary arm of the instrument above the first timing mark.
4. With the sample flow freely, measure in seconds to within 0.1s, the time required for the meniscus to pass from the first to the second timing mark.
5. Repeat to procedure describes in 6.2.4 to make a second measurement of flow time.
6. Calculate kinematic viscosity,ν, in mm^2/s from each measured flow time.
7. If the two determinations of kinematic viscosity, calculated from the flow time measurements, agree within the stated repeatability for product, use the average of these determinations to calculate the kinematic viscosity result to be reported.

- Calculation

Calculation of the kinematic viscosity is carried out using the following equation A1.

$$\nu = c \times t \qquad (A1)$$

Where, ν = kinematic viscosity in mm^2/s

c = calibration constant of the viscometer in $(mm^2/s)/s$ unit

t = measured flow time in second unit

- Precision

Repeatability

The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in long run, in the normal and correct operation of this test method.

Base oil at 40 and 100 °C	$\pm$ 0.0011x
Formulated oil at 40 and 100 °C	$\pm$ 0.0026x

Where, x is the average of results being compared

A2. Flash point (ASTM D-92) [12]

The flash point of an oil is the lowest temperature at which it gives off vapors that will ignite when a small flame is periodically passed over the surface of the oil.

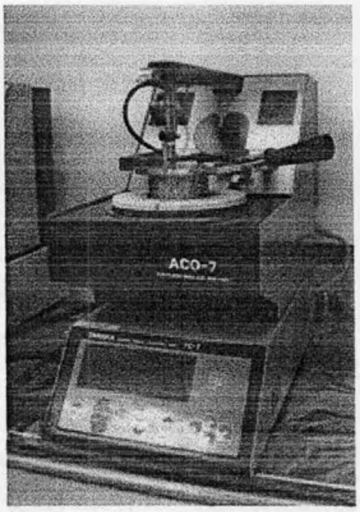


Figure A2 Flash point apparatus

- Purpose

To determining the flash point of oil via petroleum product by Cleveland Open Cup apparatus (Fig. A2).

- Definition

Flash Point is the lowest temperature corrected to a barometric pressure of 101.3 kPa (760 mm Hg), at which application of an ignition source causes the vapors of a test specimen of sample to ignite under specified conditions of test.

- Scope of test

The Cleveland cup is filled with oil sample up to the specified filling mark. The bulb of the thermometer is immersed in the sample ¼ inch from the bottom of the cup. The oil is then heated at a constant rate. At every 2 °C rise in temperature a small flame is passed over the oil surface. When a flash occurs, the temperature reading is the flash point of the sample.

Remark : This test method is applicable to all lubricating oils with flash points above 70 °C and below 400 °C.

- Test method

General Procedure for flash point

1. Wash the test cup with solvent to remove any test specimen or traces of gum or residue remaining from a previous test. If any deposits of carbon are present, they should be removed with a material such as silicon carbide grit paper.
2. Ensure that the test cup is completely clean and dry before using again.
3. Ensure that the air ventilation by fume hood is stopped. The air flow can influence the vapors in the test cup and test flame.

Procedure for Transparent Liquids

1. Fill the test cup with approximately 70 ml of oil sample or reach to a level mark inside the cup, and place the test cup on the center of the heater. The temperature of the test cup and sample shall not be exceed 56 °C below the expected flash point.
2. Start the automated apparatus and adjust the test flame to a diameter of 3.2 to 4.8 mm or to the size of the comparison bead which is mounted on the apparatus.
3. Record data which shown on display when occurred flash point.

- Precision

Repeatability

The difference between successive results, obtained by the same operator with the same apparatus under constant operating conditions on identical material, would in the long run, in the normal and correct operation of the test method.

Flash Point ± 8 °C

A3. Total acid number (ASTM D-664) [13]

This method determines the acidic constituents in a petroleum oil that are soluble in mixtures of isopropyl alcohol and toluene. It may be used to show relative changes occurring in an oil under oxidizing conditions.

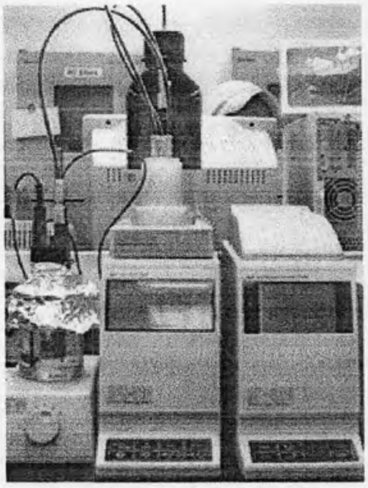


Figure A3 automatic potentiometric titrator apparatus

- Purpose

To determine the total acid number in oil sample by use automatic potentiometric titrator (Fig. A3).

- Definition

Total acid number (TAN) is the quantity of base, expressed in milligram of potassium hydroxide, that is required to titrate all acidic constituents present in 1 g of samples.

Furthermore, total acid number is shown relative changes in an oil due to oxidation. Comparing the TAN with the values of a new oil will indicate the development of harmful products or the effect of additive depletion.

- Scope of test

This test method is the determination of acidic constituents in petroleum products and lubricants soluble or nearly soluble in mixtures of toluene and 2-propanol. By a weighed amount of sample in titration solvent is titrated with a standard alcoholic potassium hydroxide solution to a definite end point.

Remark : It is applicable for the determination of acids whose dissociation constants in water are larger than 10^{-9}; extremely weak acids whose dissociation constants are smaller than 10^{-9} do not interfere.

- Test method

1. At first time of using machine should prepare for blank test by fresh titration solution 125 ml into titration beaker.
2. Drop magnetic bar into titration beaker and set up electrode. And then start machine to detect the end point.
3. When finished the test, remove mixture remained at electrode by soft wiper and rinse the electrode with alcoholic solvent and DI water then dip the electrode into DI water for 5 minutes before next sample.
4. Weighing sample about 20 ± 0.5 g into 250 ml titration beaker then mix with 125 ml of titration solution.
5. Record the result as total acid number.

- Calculation

Quantitative

The total acid number or acidity of hydro oil sample is determined by automatic calculation of machine when finished the titration.

Calculation formula

$$\text{Acid number (mg KOH/g)} = (A - B) \times M \times 56.1 / W \qquad \text{(A2)}$$

Where, A = alcoholic KOH solution used to titrate sample to end point in ml unit

B = volume corresponding to A for blank titration in ml unit

M = concentration of alcoholic KOH solution in mol/l unit

W = sample mass in gram unit

A4. Moisture content (ASTM D-1744-92) [14]

The Moisture content or water content of petroleum products can be useful to predict quality and performance characteristics of the product.

Figure A4 Karl Fischer Titrator apparatus

- Purpose

To analyze the moisture content by using Karl Fischer Titrator (Fig. A4).

- Definition

Moisture content of petroleum product can be useful to predict quality and performance characteristics of the products. The material to be analyzed is titrated with standard Karl Fischer reagent to an electrometric end point.

- Scope of test

This test method covers the determination of moisture content in liquid petroleum products.

- Test method

1. Should prepare for blank test by titration without sample injection.
2. Weighing 1 ml of oil sample with glass syringe and input weight to sample detail.
3. Inject sample to titration cell and push start to titrate.
4. After injected sample, weighing the remaining sample with glass syringe again and input weight to sample detail.
5. Record the result as the moisture content without any correction.

- Calculation

The moisture content result must be multiply to a constant that come from performance check by standard water solution as equation A3.

$$\text{moisture content} = (CF \times 1{,}000) / W \qquad (A3)$$

Where,
C = milliliters of reagent required for tritration of the sample
F = water equivalence in ml unit
1,000 = factor for converting to parts per million
W = weight of sample used in grams unit

- Precision

Repeatability

The difference between successive results, obtained by the same operator with the same apparatus under constant operating conditions on identical material, would in the long run, in the normal and correct operation of the test method.

Water content (ppm)	repeatability (ppm)
50 to 1,000	± 0.1 x

Where, x denotes average result

A5. Specific gravity (ASTM D-4052) [15]

The specific gravity is a fundamental physical property that can be used in conjunction with other properties to characterize both the light and heavy fractions of petroleum and petroleum products.

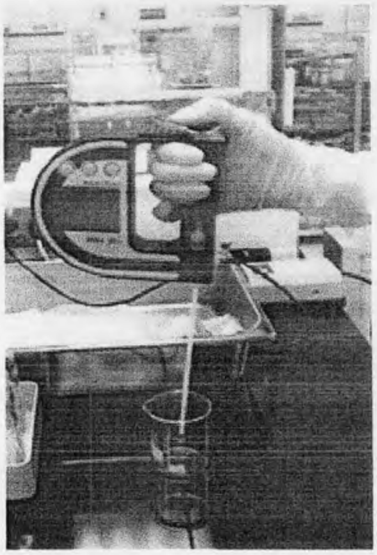


Figure A5 Digital density analyzer

- Purpose

This test method covers the determination of specific gravity of petroleum distillates and viscous oils that can handed in a normal liquids at test temperatures between 15 and 35 °C by Digital density analyzer (Fig. A5). Its application is restricted to liquids with vapor pressure below 600 mm Hg (80 kPa) and viscosities below about 15,000 mm^2/s at temperature of test.

- Definition

Specific gravity is the ratio of the density of a material at a stated temperature to the density of water at a stated temperature.

- Scope of test

A small volume (approximately 0.7 ml) of liquid sample is introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in the mass of tube is used in conjunction with calibration data to determine the specific gravity of the samples.

- Test method

1. Flush the sample tube by the sample for 3 times then introduce a small amount of sample into sample tube of the instrument. Make sure that no bubbles are trapped in the tube.
2. After the instrument displays a steady reading to four significant figures for density, indicating that temperature equilibrium has been reached, record the density.

- Precision

Repeatability

The different between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty.

Range	Repeatability
0.68 – 0.64	$\pm$ 0.0001

A6. Pour point (ASTM D-5950) [16]

The pour point of a petroleum oil is the lowest temperature at which the oil will pour or flow under prescribed conditions when it is chilled without disturbance at a fixed rate. Moreover, pour point is of importance in establishing the lowest temperature at which a diesel fuel is still sufficiently fluid to be pumped or transferred. However, many fuels can be pumped or transferred at temperatures below their pour point.

The pour point of petroleum product is an index of the lowest temperature of its utility for certain applications. Flow characteristics, like pour point, can be critical for the correct operation of lubricating oil system.

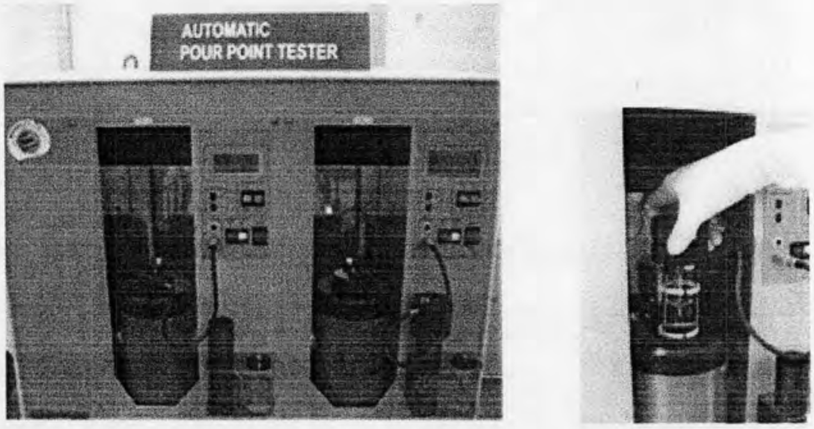


Figure A6 automatic pour point apparatus

- Purpose

This test method covers the determination of pour point of petroleum products by an automatic instrument that tilts the test jar during cooling and detects movement of the surface of the test specimen with and optical device as Fig. A6.

Remark : This test method includes the range of temperatures from -60 to +60 °C.

- Definition

Pour point is the lowest temperature at which movement of the test specimen is observed under the prescribed conditions of this test method.

- Scope of test

After preliminary heating, the test specimen is inserted into the automatic pour point apparatus. After starting the program, the specimen is cooled according to the cooling profiled. The lowest temperature at which movement of specimen is detected, by the automatic equipment, is displayed as the pour point.

- Test method

1. Pour the sample into the test specimen jar to the level mark. When necessary, heat the sample in a water bath or oven until it is just sufficiently fluid to pour the sample into the specimen jar.
2. Place the test jar in the selected test cell. Attach the detector head according to the manufacturer's instruction.
3. Enter the expected pour point.
4. Start the test in accordance with the manufacturer's instruction.
5. At this point, the instrument shall monitor the test specimen with the optical detector, adjusting the jacket temperature to the first temperature level and measuring the specimen temperature. When the pour point is determined, the instrument shall display the pour point result and start to reheat the test specimen.
6. Record the result as the pour point without any correction.

- Precision

Repeatability

The difference between successive test results, obtained by the same operator using the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of this test method, exceed the following, only in one case in twenty.

$\pm$ 4.4 °C

A7. Corrosive outgas test by Copper (Cu) - Silver (Ag) coupon method

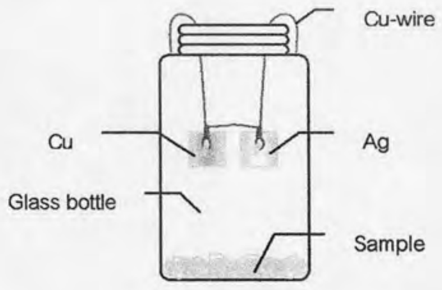


Figure A7 Illustration of sample preparation of corrosive outgas test by Cu- Ag coupon

- Purpose

This test method to measure the corrosive outgas of lubrication oil on copper and silver coupon under high humidity condition.

- Scope of test

Hang a copper and silver coupon by copper wire over the sample at center of bottle and then test it at condition of test as 70 °C at 90%RH for 24 hours. At the end of test period, remove sample from the oven and allow them to cool to room temperature. Take photo of both coupons after that analysis the composition of surfaces by SEM-EDX.

- Test method

Copper and silver coupon preparation

1. Cut copper and silver strip to coupon size around 0.8 x 1.0 cm.
2. Immerse coupon in acid solution, nitric acid [1] : DI water [1], about 30 seconds or the surface of both coupons are rough.
3. Immerse coupon in DI water 4 times.
4. Immerse coupon in Acetone 1 time.
5. Allow coupon to dry at room temperature.

Method

1. Fill sample in clean bottle, quantity of sample around 1 g.
2. Hang a copper and silver coupon by copper wire over the sample at center of bottle.
3. Cover the bottle with aluminum foil and seal by Teflon tape then make 8 holes at aluminum foil around rim of bottle.

4. Place prepared bottle in oven, condition of test as 70 °C at 90%RH for 24 hours.
5. At the end of test period, remove sample from the oven and allow them to cool to room temperature.
6. Take photo of both coupons after that analysis the composition of surfaces by SEM-EDX.

A8. Corrosion test by Copper plate method (ASTM D-130) [17]

It measures the corrosive attack of liquid petroleum products on copper by immersion under static laboratory conditions. The extent of damage is classified by comparing appearance with ASTM standards.

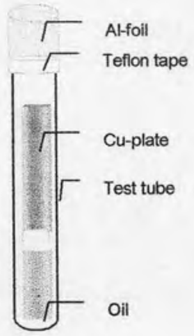


Figure A8 Illustration of sample preparation of corrosion test by Copper plate method

- Purpose

This test method to measure the corrosive attack of lubricating oil on copper plate under static laboratory conditions.

- Scope of test

A polished copper strip is immersed in oil and heat to 100 °C for 24 hours. The condition of the test strip is compared to ASTM corrosion standards (Fig. A9 and Table A1), provided by ASTM. The test serves as a measure of oil's tendency to corrode copper under static conditions.

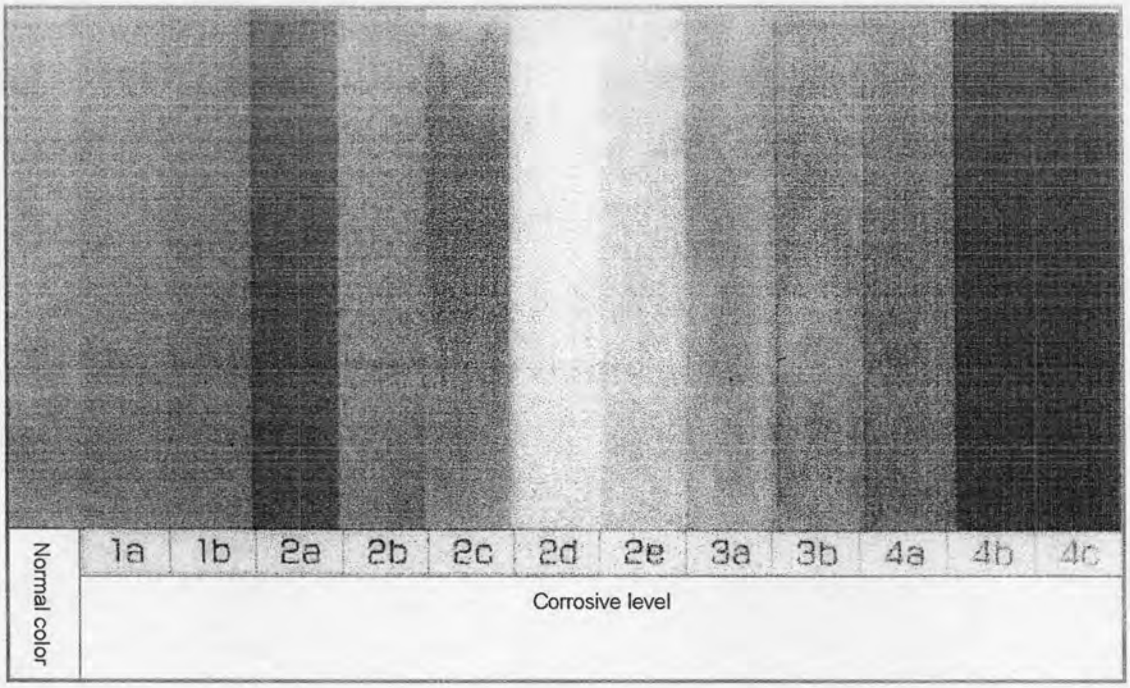


Figure A9 ASTM corrosion standards

- Test method

Copper plate preparation

1. Remove all surface blemishes from all 6 sides of the copper plate with silicon carbide paper of such degrees of fineness as are needed to accomplish the desired results efficiently. Finish with 240-grit silicon carbide paper or cloth, removing all marks that may have been made by other grades of paper used previously.
2. Immerse the copper plate in Acetone which it can be withdrawn immediately for final preparation (polishing) and it can be stored for future use.
3. Remove a copper plate from the wash solvent. Subsequently handle only with stainless steel forceps; do not touch with the fingers and place it on clean paper.

Method

1. Pour 3 ml oil in test tube and dip copper plate in that tube then cover tube with Aluminum foil and seal with Teflon tape.

2. At the end of the test period, remove the sample from oven and allow them to cool to room temperature. Clean copper plate by slightly rub with clean room wipers.
3. Observed the discoloration of copper plate and interpret the corrosiveness of the sample accordingly as the appearance of the test strip agrees with one of the ASTM Copper Corrosion Standards.
4. When a copper plate is in the obvious transition state between that indicated by any two adjacent standard, judge the sample by the more tarnished standard. Should a copper plate appear to have a darker orange color than standard 1b, consider the observed plate as still belonging in classification 1; however, if any evidence of red color is observed, the observed plate belongs in classification 2.
5. A claret red plate in classification 2 can be mistaken for a magenta overcast on brassy plate in classification 3 if the brassy underlay of the latter is completely masked by a magenta overtone. To distinguish, immerse the plate in wash solvent; the former will appear as a dark orange plate while the latter will not change.
6. Take photo for discoloration of copper plate by microscope.

Table A1 Description of ASTM corrosion standards [18]

Classification	Designation	Description
1a	Slight tarnish	Light orange, nearly identical to freshly polished strip
1b	Slight tarnish	Dark orange
2a	Moderate tarnish	Claret red
2b	Moderate tarnish	Lavender
2c	Moderate tarnish	Multicolored with lavender blue or silver, or both, overlaid on claret red
2d	Moderate tarnish	Silver
2e	Moderate tarnish	Brass or gold
3a	Dark tarnish	Magenta overcast on brass strip
3b	Dark tarnish	Multicolored with red and green, but no gray
4a	Corrosion	Transparent black, dark gray or brown, with some green barely showing
4b	Corrosion	Graphite or lusterless black
4c	Corrosion	Glossy or jet black

A9. Weight loss by TG/DTA instrument

Thermo gravimetric (TG) is the instrument for thermal analysis. Sample mass is gradually decreased when heating is applied to sample continuously. Measurements of changes in sample weight with temperature are using thermo balance. A thermo balance is a combination of a furnace, a temperature programmer and computer for control and data capture.

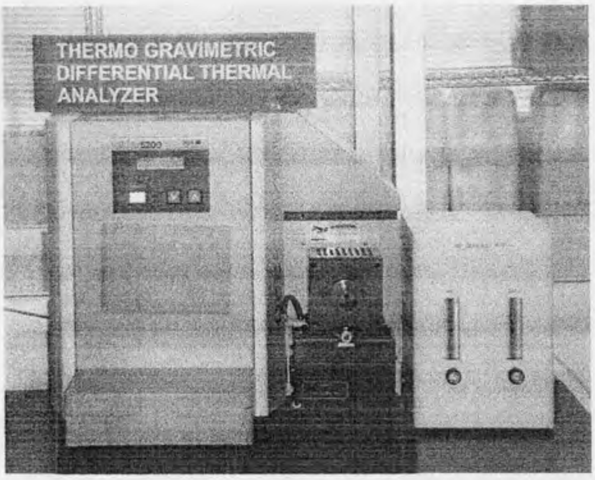


Figure A10 TG/DTA instrument

- Purpose

Monitoring of the temperature difference between a sample and an inert reference as they are heated uniformly. Endothermic or exothermic changes in the sample lead to characteristic deviations in temperature.

- Scope of test

When sample weight changed, the optical position sensors detect changes in the position of slit and send the signal to balance circuit. The balance circuit feedback current for return the position of slit to balance. The current is detected and converted into weight signals.

- Test method

1. Turn on the power to the station.
2. Turn on the power to the TG/DTA module and wait a while, and then check that the liquid crystal displays shows "N Ready" (N is the channel no. it is connected to).
3. If gas flow during measurement is require, start the gas flow in advance.
4. Display the measurement screen for the Channel number connected to the TG/DTA module. Sample weighting is performed using the following as weight of sample about 10 mg into aluminum pan.
5. Pull the furnace to the front, turn off the fan switch, and place the containers that set program as bake at temperature 150 °C with rate 5.0 °C /min and hold 24 hours.

6. Close the furnace and wait for TG signal to stabilize. When it has stabilize, press zero key for the channel connected to the TG/DTA.
7. Check that the sample information of that channel read 0.0 mg.
8. Open the furnace again and place the samples in the respective pans, close the furnace.
9. Again wait for the TG signal to stabilize.
10. Go to the sample window. When the signal stabilize, move the cursor to AUTO WEIGHT and press enter.
11. Start run.
12. After finished testing, weight loss was record.

A10. Outgas analysis by GC/MS instrument

Identification of outgases compounds is performed automatically by operating software. These results are then verified manually by the operator. The concentrations of the outgases compounds are the calculated according to the following equation A4.

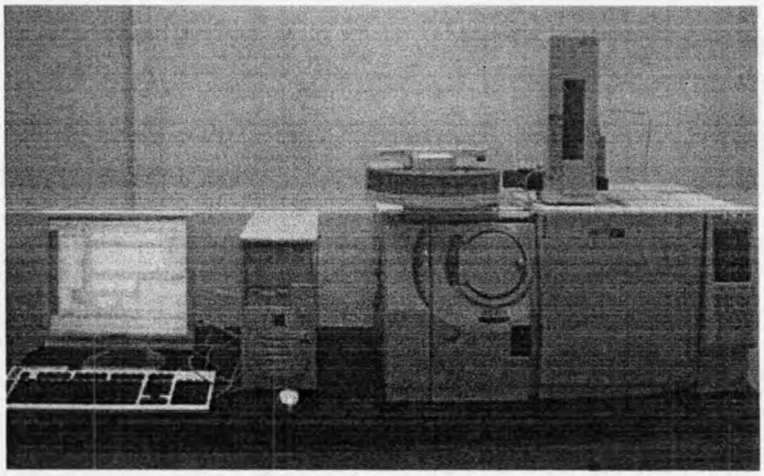


Figure A11 GC/MS instrument

$$[A] = [S^*] \times \text{Area sample} / \text{Area}^* \times \text{mg of sample} \qquad \text{(A4)}$$

Where, [A] is the concentration of outgassed compound in ng unit

[S]* is the concentration of the internal or external standard in ng unit

Area sample is the peak area for the outgassed compound

Area* is the peak area of the internal or external standard

- Test method

Sample preparation

1. Draw sample oil by use micropipette and fill in aluminum oil pan after that weight sample approximate 5-6 mg.
2. Insert adsorption tube which contain Absorption Material (Carbotrap B&C) to the holder of oven that set oven to 85 °C and then place the sample after weight in the chamber and latch it to secure the seal. Start the nitrogen flow into chamber 50 ± 5 ml/min, after 3 hours remove the adsorption tube from the holder.

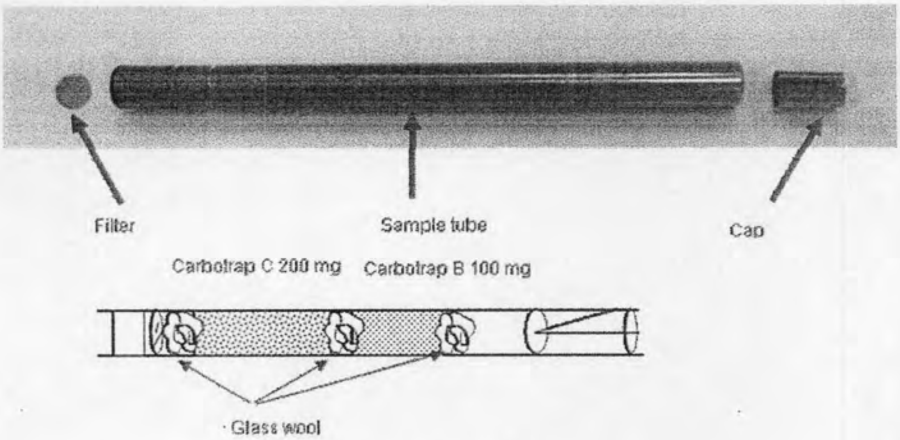


Figure A12 Adsorption tube

3. Place adsorption tube as Fig.A12 into the ATD to desorb the tubes for 10 minutes at 380 °C. Program the ATD to desorb the tubes as described in table A2. The desorbed material will be automatically injected into GC-MS by set the condition of ATD and GC as follow table A2.

Table A2 Condition of GC analysis

Parameter	Condition
Baking condition	85 °C
Nitrogen gas flow rate	50 ± 5 ml/min
Chamber	Teflon chamber
1 °Absorption Material	200 Carbotrap C + 100 Carbotrap B
ATD400 condition	Sample desorb temp. = 380 °C Coolant temp. = - 30 °C Desorp time = 10 minutes Desorb Flow = 50 ± 0.5 ml / min Outlet Split flow = 25 ± 0.5 ml/min ATD pressure = 16.2 ± 0.1 psi
GC-MS Condition	
Oven program	
- Initial temp	40 °C for 2 minutes
- Ramp.	8 °C / min
- Final temp.	240 °C for 13 minutes
Analysis mode	Split 15:1
Interface temp.	280 °C
Column	XTI-5 (30.0m x 0.25 mm x 0.25 um)

A11. Wear analysis by Four wear ball tester (ASTM D-2266) [19]

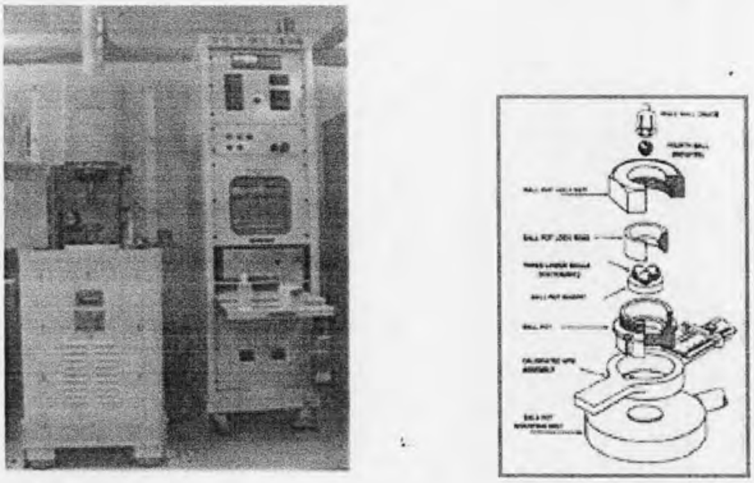


Figure A13 Four wear ball tester

- Purpose

This test method covers the determination of the wear preventive characteristics of oil lubricant in sliding steel-on-steel applications. It is not intended to predict wear characteristics with metal contaminations other than steel-on-steel or to evaluate the extreme pressure characteristics of the oil lubricant.

- Scope of test

The determination of the load carrying capacity of a lubricant in kilograms applied to a system of four steel balls in the form of a tetrahedron.

The four ball wear tester consists of 4 balls arranged in the form of an equilateral tetrahedron. The basic elements are 3 lower balls held immovable in a clamp to form a cradle in which a fourth or upper ball is rotated around a vertical axis under prescribed conditions of load and speed. Usually, the balls are made of steel, but a disc adapter is furnished for making wear measurements of other materials.

The points of contact are lubricated by immersion in the cup surrounding the four ball assembly. During the test, circular scars are worn on the surface of the 3 load, stationary balls; the average diameter of the scars, a measure of wear, depends on the speed, duration of test and character of the lubricant.

- Test method

1. Place one of the clean steel test balls into the chuck. Tighten the chuck holding the test ball firmly onto the shaft of the machine.
2. Insert the 3 test balls in the ball pot and lock the balls in position by tightening the lock nut on the ball pot.
3. Coat the test balls located in the chuck and pot completely and thoroughly with the test oil; fill the ball pot with oil and level off with the top surface of the locknut.
4. Set to four ball wear machine.
5. Set up testing program as follow below parameter and start test.

   Test parameter

   Temperature : 75 °C
   Speed : 1,200 ± 50 rpm
   Load : 40 ± 0.2 kgf
   Duration of test : 60 ± 1 minutes

6. After a period of 60 minutes turn off the drive motor and heater. Lower the assembly and remove the ball pot.
7. Measure the scar diameter.
8. Remove the locknut and release the test balls. Clean the balls with solvent and wipe dry. Place the individual balls on a holder and by means of a microscope, measure the scar diameters at two positions on each of 3 test balls. Measure the nearest 0.01 mm the minimum scar diameter. Again to the nearest 0.01 mm, measure the minimum scar diameter 90° from the first position of the measurement.

- Precision

Repeatability

The difference between two test results, obtained by the same operator with same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty.

$\pm 0.1x$ mm.

Where, x denotes average result

A12. Friction coefficient analysis by SRV (ASTM D-5707) [20]

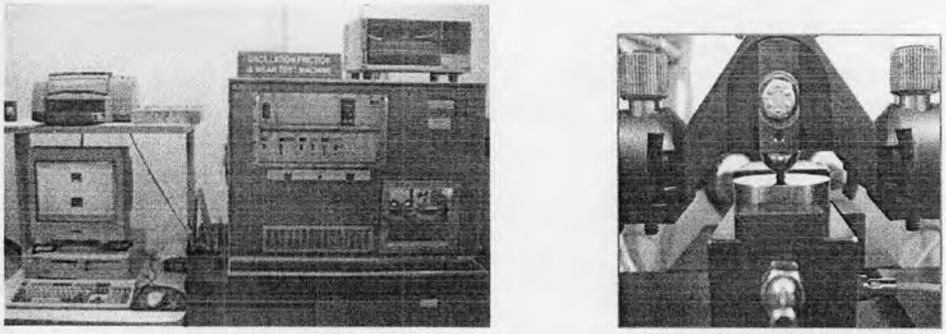


Figure A14 SRV instrument

- Purpose

To analyze friction coefficient of lubricant by Oscillation Friction and Wear test machine (Fig. A14). Its ability to protect against wear when subject to high-frequency, linear-oscillation motion using.

- Definition

Coefficient of friction is the dimension less ratio of the friction force (F) between two bodies to the normal force (N) pressing these bodies together.

- Scope of test

This test method for determining a lubricating oil's coefficient of friction and its ability to protect against wear when subjected to high-frequency, linear-oscillation motion using an SRV test machine at test load of 200N, frequency of 50 Hz, stroke amplitude of 1.00 mm, duration of 2 hours and temperature -20 °C and 50 °C.

- Test method

1. Clean the test pieces by sonicate with solvent for 5 minutes.
2. Set up test pieces as ball fix to holder and specimen base installed.
3. Use a spatula to place the sample on the test surface with a bit amount of oil.
4. Set load for 50 N and release load to ball to make sample touching on specimen surface.
5. Set up testing program as follow below parameter and start test.

Method for normal temperature

Test parameter

Load : 50 N

Temperature : -20 °C

Frequency : 50 Hz

Stroke : 1 mm

Time : 2 hours

Method for low temperature

Test parameter

Load : 50 N

Temperature : 50 °C

Frequency : 50 Hz

Stroke : 1 mm

Time : 2 hours

- Acceptance Criteria

Acceptance Criteria of grease and oil's friction coefficient do not exceed 0.3.

- Precision

Repeatability

The difference between two test results, obtained by the same operator with same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty.

$$\pm 0.1\ x$$

Where, x denotes average result

A13. Oxidation induction time of lubricant by RBOT (ASTM D-2272) [21]

The instrument use to utilizes an oxygen-pressured vessel to evaluate the oxidation stability of oil sample in the presence of water and copper catalyst coil at high temperature

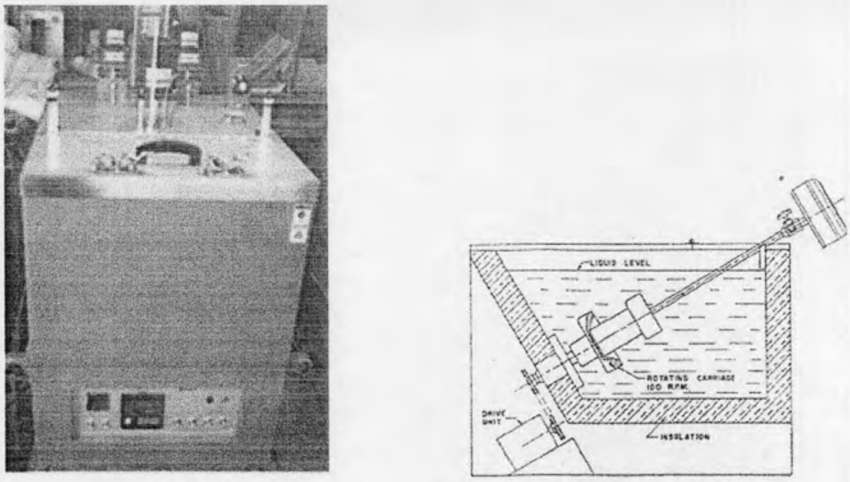


Figure A15 RBOT instrument

- Purpose

The estimate of oxidation stability is useful in controlling the continuity of this property for batch acceptance of production lots having the same operation. Furthermore, it is also used to assess the remaining oxidation test life of in-service oils.

- Scope of test

The test oil, water and copper catalyst coil, contained in a covered glass container, are placed in a bomb equipped with pressure gauge. The bomb is charge with oxygen to pressure of 620 kPa, paced in a constant temperature oil bath set at 150 °C and rotated axially at 100 rpm at an angle 30 degrees from horizontal. The time for the test oil to react with given volume of oxygen is measured, completion of the time being indicated by a specific drop in pressure of 175 kPa.

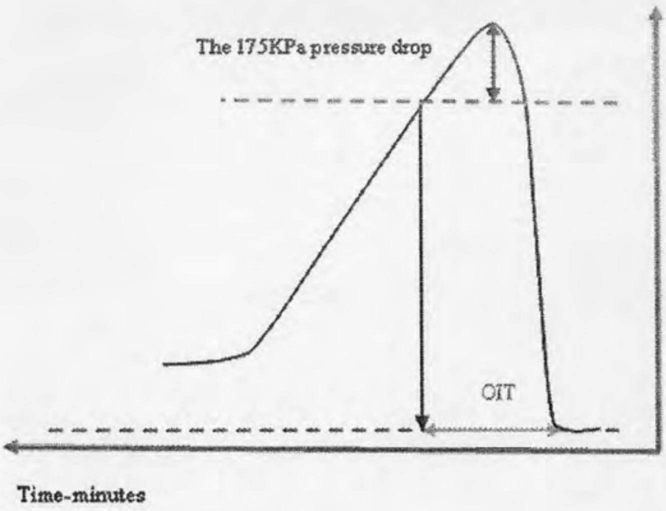


Figure A16 Oxidation induction time (OIT) by RBOT instrument

From Fig. A16, observe the plot of the recorded pressure versus time and pressure. The result (Oxidation induction time) will be recorded the time at the point on the falling part of the curve where the pressure is drop 175 kPa from the highest pressure.

- Test method

Cu catalyst coil preparation

1. Before use, must be polish approximately 3 m of copper (Cu) wire with the silicone carbide abrasive cloth and wipe free from abrasive with the clean, dry cloth.
2. Wind the wire into a coil having an outside diameter 44 to 48 mm and stretched to a height of 40 mm to 42 mm.
3. Clean the coil thoroughly with solvent and make dry.

Sample preparation

1. Insert freshly cleaned catalyst coil inside the glass sample container by turning motion.
2. Set the weight of the glass sample container with a freshly cleaned catalyst coil to zero.
3. Weight 50 ± 0.5 g. of sample oil into the container; also add 5 ml of DI water.
4. Add another 5 ml of DI water to the vessel body.
5. Slide the sample container into vessel body.

6. Cover the glass container with 57.2 mm PTFE disk and place a PTFE ring on top of the PTFE disk .
7. Apply a thin coating of silicone grease to the O-ring vessel seal located in the gasket groove of the vessel cap to provide lubrication.
8. Insert the cap into the vessel body.
9. Tighten the closure ring. Cover the threads of the oxygen pressure sensor.
10. Attach the oxygen line with an inline pressure gage to the inlet valve on the vessel stem.
11. Slowly turn on the oxygen supply valve until the pressure has reached 620 kPa.
12. Turn off the oxygen supply valve. Slowly release pressure by purging process two more times; purge step should take approximately 3 minutes.
13. Adjust the regulating valve on the oxygen supply tank to 620 kPa at room temperature, 5 kPa shall be added or subtracted to attain the required initial pressure.
14. Fill the vessel to this required pressure and close the inlet valve securely by hand.

Method

1. Before testing, bring the heating bath to the test temperature while the stirrer is in operation.
2. Switch off stirrer, insert the vessel into the carriages and record the time.
3. Restart the stirrer.
4. The bath temperature shall stabilize at the test temperature within 15 minutes after the vessel is inserted. Maintain the test temperature within ± 0.1 °C.
5. Keep the vessel completely submerged and maintain continuous and uniform rotation throughout the test. A standard rotational speed of 100 ± 5 rpm is required.
6. The test is complete after the pressure drops more than 175 kPa below the maximum pressure.

7. After termination of the test, the vessel shall be removed from the oil bath and cooled to room temperature.

- Precision

Repeatability

The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in long run, in the normal and correct operation of this test method.

± 30 minutes

A14. Evaporation loss

Evaluates the potential for evaporation loss of lubricant components in high temperature service such as bake in control temperature oven. Evaporation loss is measured by the change in sample weight during the test.

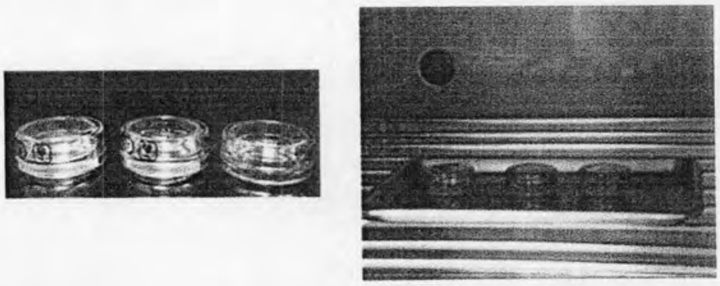


Figure A17 Evaporation loss test

- Test method

1. Weight oil sample about 3 ± 0.5 g into Petri dish.
2. Bake oil at testing temperature as 60 °C and 120 °C for 3 months by each month, weight loss of sample are recorded.
3. After record weight, oil sample will be put into oven again for continue test at same started testing temperature.

A15. Compatibility test between lubricants and bearing component

Compatibility is the investigation of effect of oil to bearing part under high temperature as shown in Fig. A18. After the test is finished, oil is assessment in the term of chemical composition by FTIR technique and total acid number. Surface analysis by microscope and SEM/EDX will be performed for bearing part.

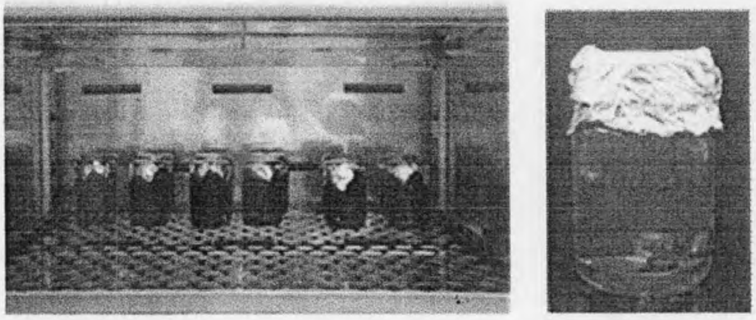


Figure A18 Compatibility test

- Test method

1. Put bearing part into glass bottle and then poured 40 ml of oil sample into diameter 3.3 cm. x Height 6 cm. of glass bottle.
2. Bake oil sample at 60 °C and 120 °C for 1 month, 2 months and 3 months, respectively.
3. After finished for each condition, oil sample was analyzed about total acid number item. For bearing part will be analyzed about surface analysis by Microscope and SEM/EDX when found stain on part.

A16. Service life test

- Test method

1. Inject oil into FDB spindle motor which use for 2.5 inch HDD.
2. Testing running at speed 5,400 rpm for 3 months at 80 °C and 100 °C.
3. Record run current in mv unit of sample for each month.

APPENDIX B
ANALYSIS RAWDATA

Table B1 Wear scar by Four ball wear tester

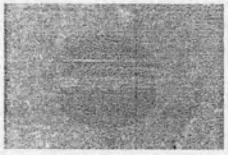
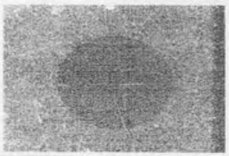
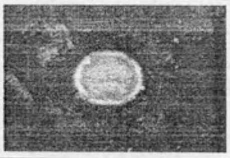
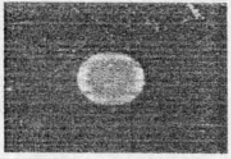
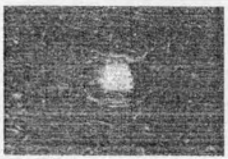
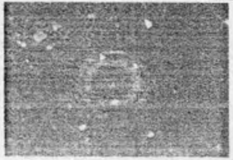
Sample	**Wear scar**	
	Scar picture	**Scar size (unit : mm.)**
	Base oil	
C		0.61
D		0.61
E		0.62
F		0.64
G		0.65
	Finished lubricant	
II		0.40
III		0.37
V		0.33
VI		0.31
Commercial B		0.30

Table B2 Evaporation loss results of finished lubricants (unit : %)

Testing condition		Finished lubricant			
		Sample II	Sample III	Sample VI	Commercial B
60 °C	1 month	0.024	0.031	0.032	0.084
	2 months	0.033	0.048	0.047	0.099
	3 months	0.045	0.053	0.055	0.105
120 °C	1 month	0.346	0.564	0.570	5.321
	2 months	1.892	2.459	2.506	11.244
	3 months	3.517	5.184	5.824	14.356

Table B3 Total acid number after compatibility test (unit : mgKOH/g)

Testing condition		Finished lubricant			
		Sample II	Sample III	Sample VI	Commercial B
60 °C	1 month	0.1383	0.2842	0.1603	0.1848
	2 months	0.2451	0.3107	0.1691	0.1897
	3 months	0.4588	0.5307	0.1895	0.2089
120 °C	1 month	0.5382	0.6378	0.1862	0.2979
	2 months	15.3201	17.6642	0.2990	0.4038
	3 months	22.5072	25.0012	0.4846	0.8393

Table B4 Running current of motor at 5,400 rpm running speed at 80 °C for 3 months (unit : mA)

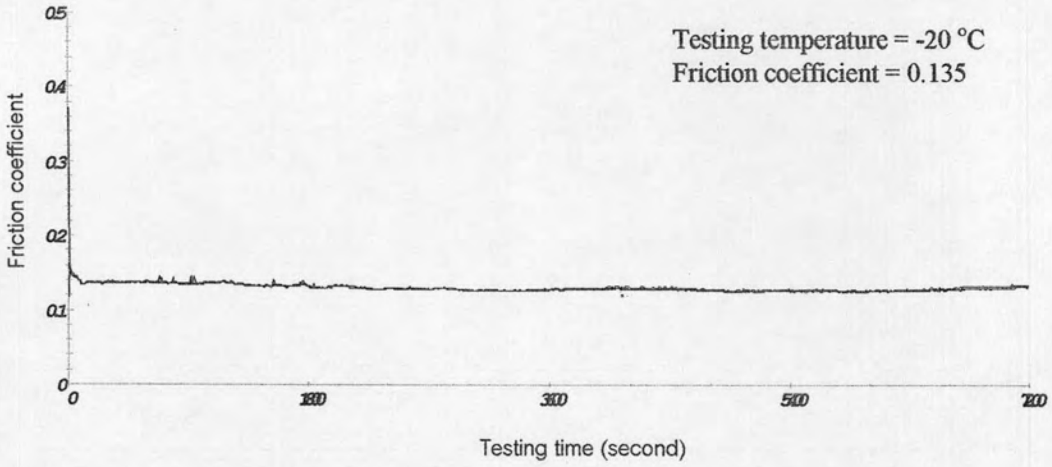
Finished lubricant		**initial**	**1 month**	**2 months**	**3 months**
Sample II	Motor no. 1	80.9	82.4	83.5	84.7
	Motor no. 2	81.1	82.3	83.5	85.2
	Motor no. 3	81.1	82.3	83.9	84.9
	Motor no. 4	81.4	82.4	83.5	85.1
	Motor no. 5	81.2	82.4	83.7	85.1
	Average	**81.1**	**82.4**	**83.6**	**85.0**
Sample III	Motor no. 6	78.2	79.2	80.7	81.8
	Motor no. 7	78.9	79.4	80.5	82.1
	Motor no. 8	78.5	79.2	80.6	82.5
	Motor no. 9	78.9	79.8	80.7	81.2
	Motor no. 10	78.4	79.4	80.4	81.7
	Average	**78.6**	**79.4**	**80.6**	**81.9**
Sample VI	Motor no. 31	80.2	81.9	82.7	84.5
	Motor no. 32	80.4	81.8	82.4	84.2
	Motor no. 33	80.4	81.2	82.7	84.7
	Motor no. 34	80.9	81.4	82.6	84.5
	Motor no. 35	80.7	81.5	82.4	83.9
	Average	**80.5**	**81.6**	**82.6**	**84.4**
Commercial B	Motor no. 11	76.9	77.4	78.2	80.9
	Motor no. 12	76.7	77.2	78.8	80.7
	Motor no. 13	75.9	77.8	77.9	81.2
	Motor no. 14	76.2	78.7	77.6	80.5
	Motor no. 15	76.8	78.6	78.7	80.7
	Average	**76.5**	**77.9**	**78.2**	**80.8**

Table B5 Running current of motor at 5,400 rpm running speed at 100 °C for 3 months (unit : mA)

Finished lubricant		**initial**	**1 month**	**2 months**	**3 months**
Sample II	Motor no. 16	81.5	83.1	85.7	87.6
	Motor no. 17	81.4	83.5	85.4	87.4
	Motor no. 18	81.2	83.0	85.4	87.8
	Motor no. 19	81.6	83.5	85.7	88.4
	Motor no. 20	81.7	83.8	86.0	88.7
	Average	**81.5**	**83.4**	**85.6**	**88.0**
Sample III	Motor no. 21	79.2	81.8	83.2	85.4
	Motor no. 22	79.4	81.4	83.4	85.8
	Motor no. 23	79.5	81.8	84.0	85.4
	Motor no. 24	79.4	81.8	84.2	86.0
	Motor no. 25	78.9	81.9	83.7	86.1
	Average	**79.3**	**81.7**	**83.7**	**85.7**
Sample VI	Motor no. 36	80.9	82.9	85.4	86.9
	Motor no. 37	80.4	82.6	85.1	87.4
	Motor no. 38	81.1	83.4	85.3	87.8
	Motor no. 39	80.6	83.1	84.2	87.1
	Motor no. 40	80.5	83.2	85.1	86.8
	Average	**80.7**	**83.0**	**85.0**	**87.2**
Commercial B	Motor no. 31	75.7	78.5	81.1	85.7
	Motor no. 32	75.9	78.3	81.5	85.9
	Motor no. 33	76.2	78.1	80.9	85.4
	Motor no. 34	76.4	77.8	80.9	85.1
	Motor no. 35	76.9	77.9	81.4	84.9
	Average	**76.2**	**78.1**	**81.2**	**85.4**

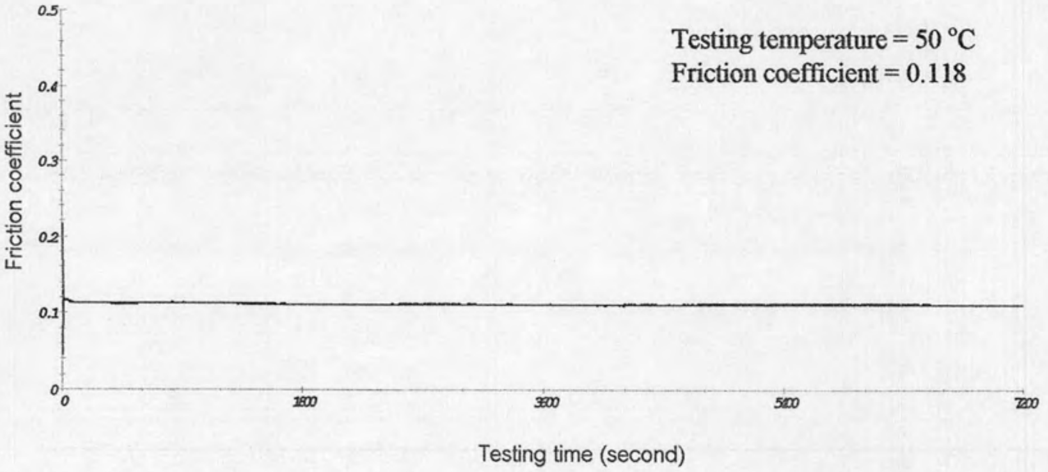
Friction coefficient by SRV instrument

Sample C



Testing temperature = -20 °C
Friction coefficient = 0.135

Figure B1 Graph of friction coefficient versus testing time at -20 °C of sample C



Testing temperature = 50 °C
Friction coefficient = 0.118

Figure B2 Graph of friction coefficient versus testing time at 50 °C of sample C

Sample D

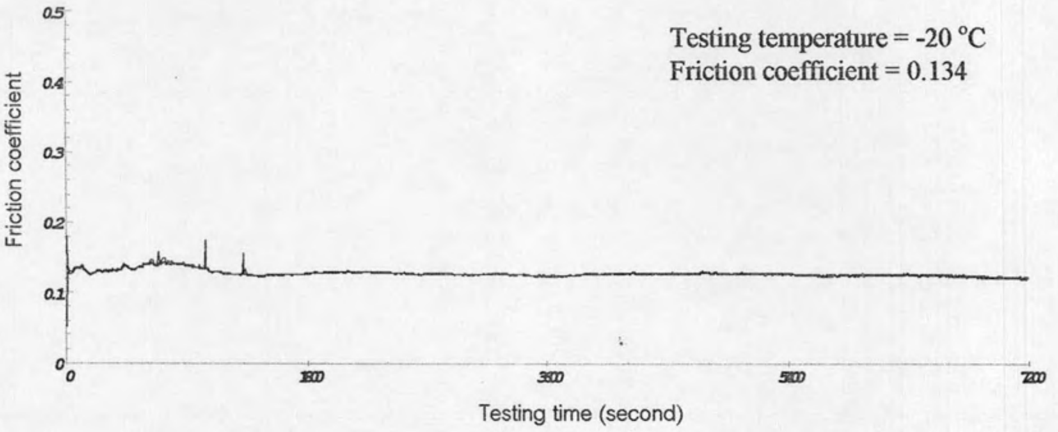


Figure B3 Graph of friction coefficient versus testing time at -20 °C of sample D

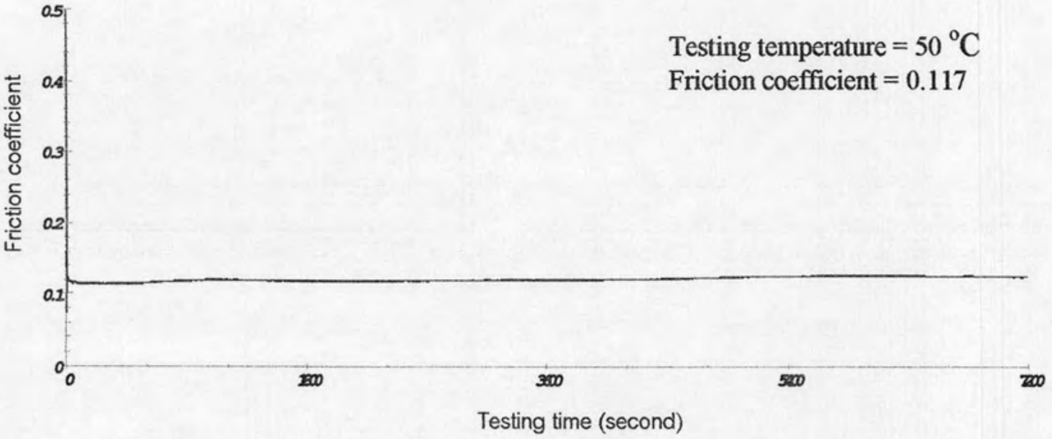


Figure B4 Graph of friction coefficient versus testing time at 50 °C of sample D

Sample E

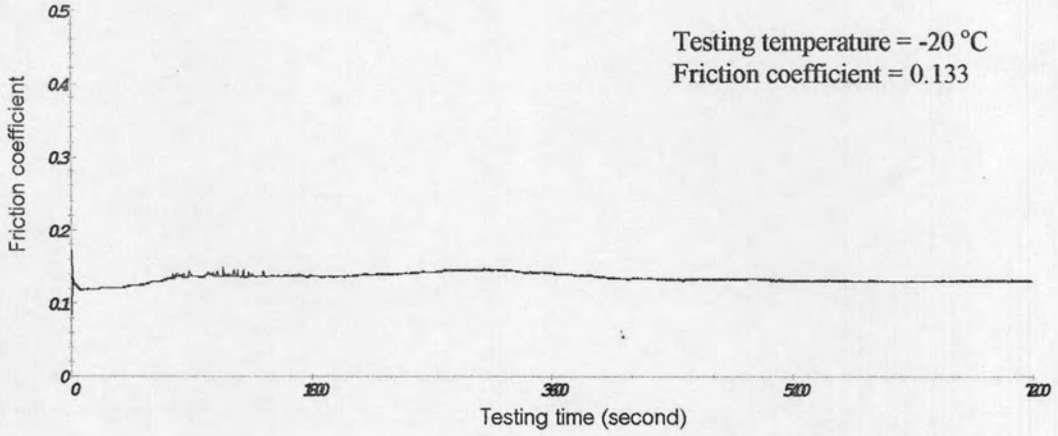

Testing temperature = -20 °C
Friction coefficient = 0.133

Figure B5 Graph of friction coefficient versus testing time at -20 °C of sample E

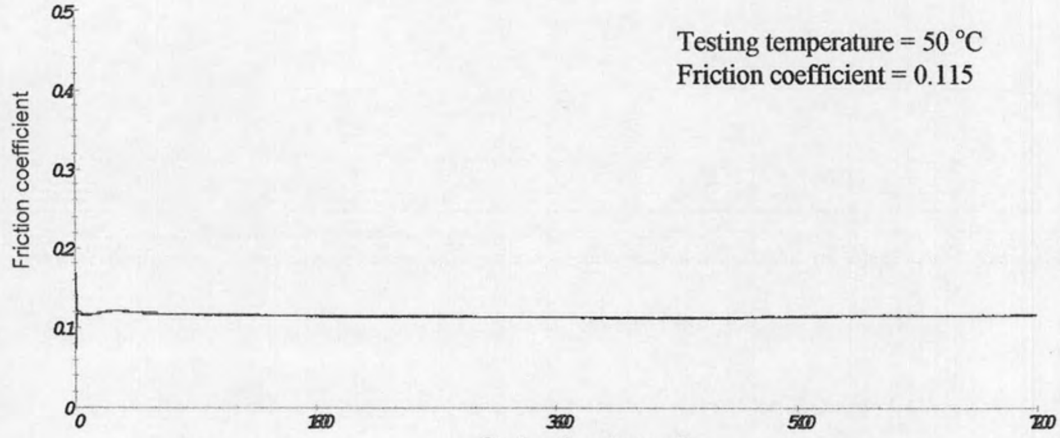

Testing temperature = 50 °C
Friction coefficient = 0.115

Figure B6 Graph of friction coefficient versus testing time at 50 °C of sample E

Sample F

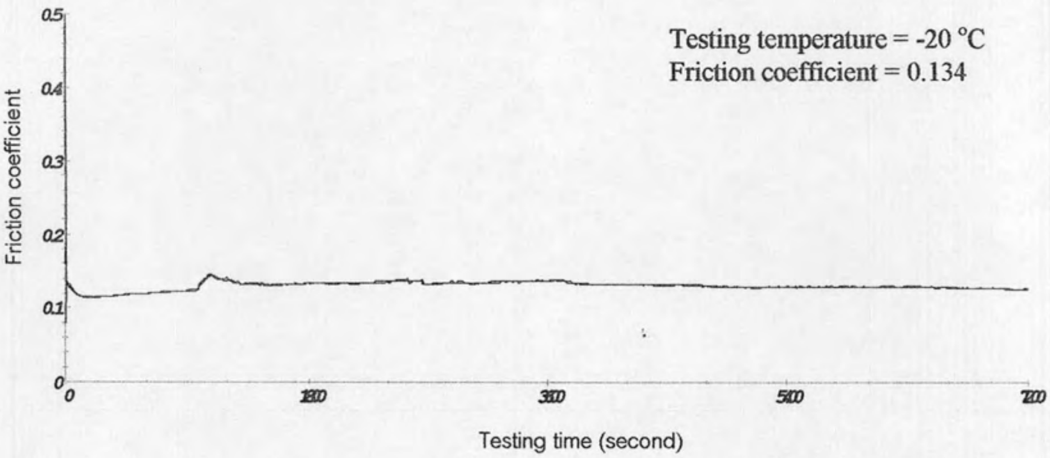


Figure B7 Graph of friction coefficient versus testing time at -20 °C of sample F

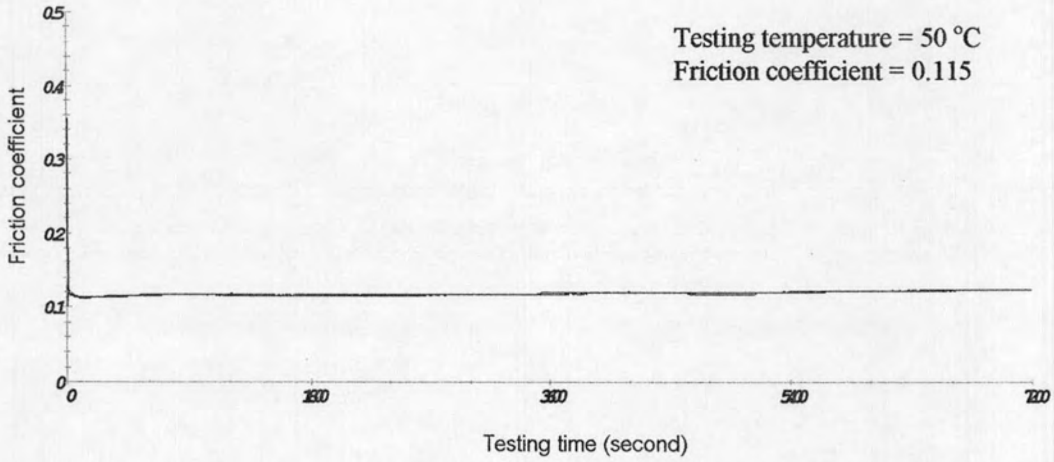


Figure B8 Graph of friction coefficient versus testing time at 50 °C of sample F

Sample G

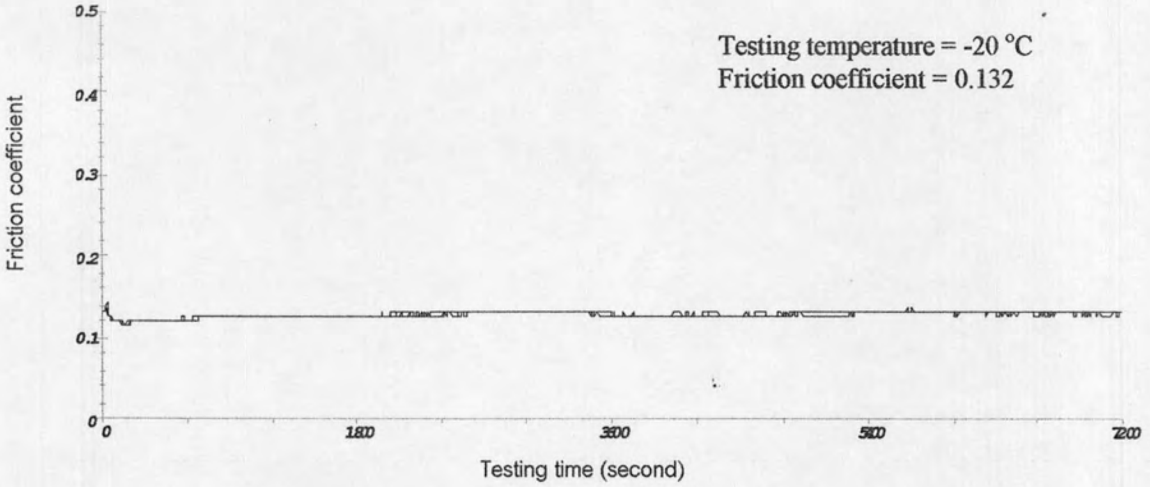


Figure B9 Graph of friction coefficient versus testing time at -20 °C of sample G

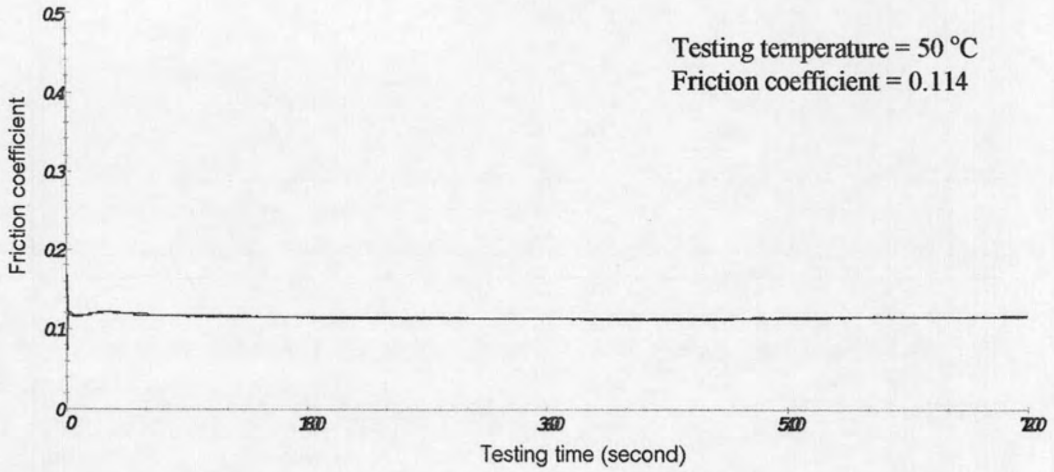


Figure B10 Graph of friction coefficient versus testing time at 50 °C of sample G

Sample II

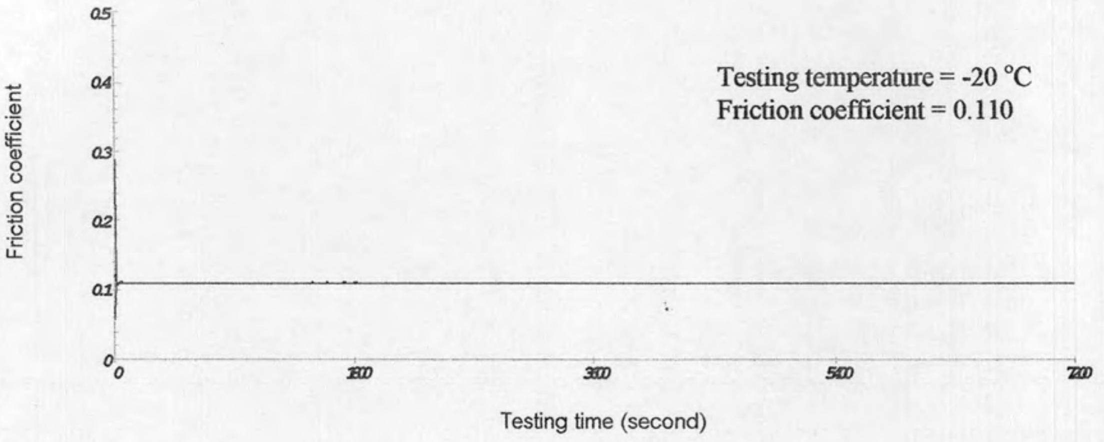


Figure B11 Graph of friction coefficient versus testing time at -20 °C of sample II

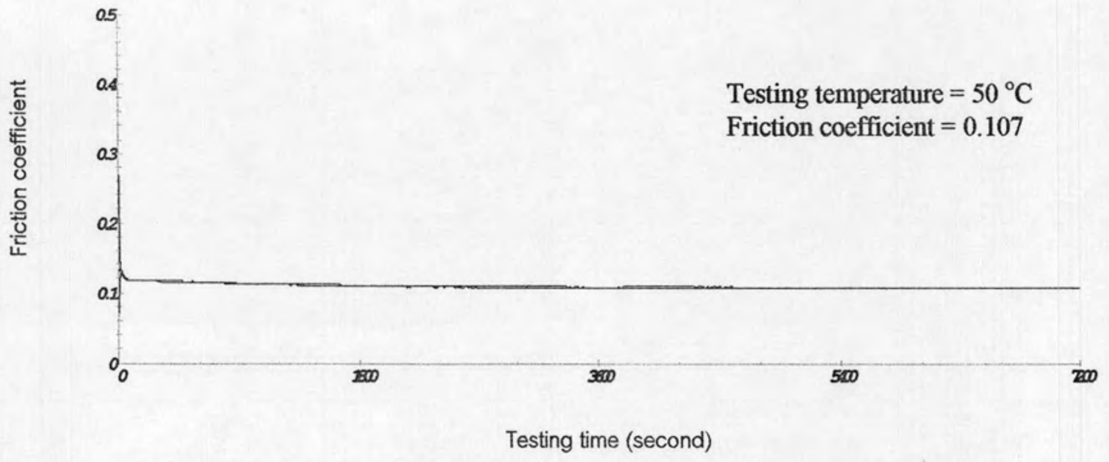


Figure B12 Graph of friction coefficient versus testing time at 50 °C of sample II

Sample III

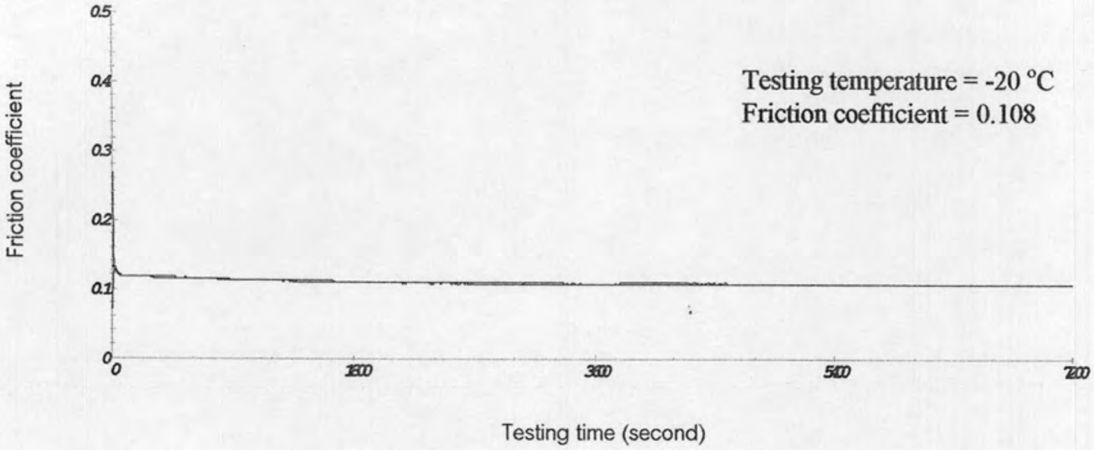


Figure B13 Graph of friction coefficient versus testing time at -20 °C of sample III

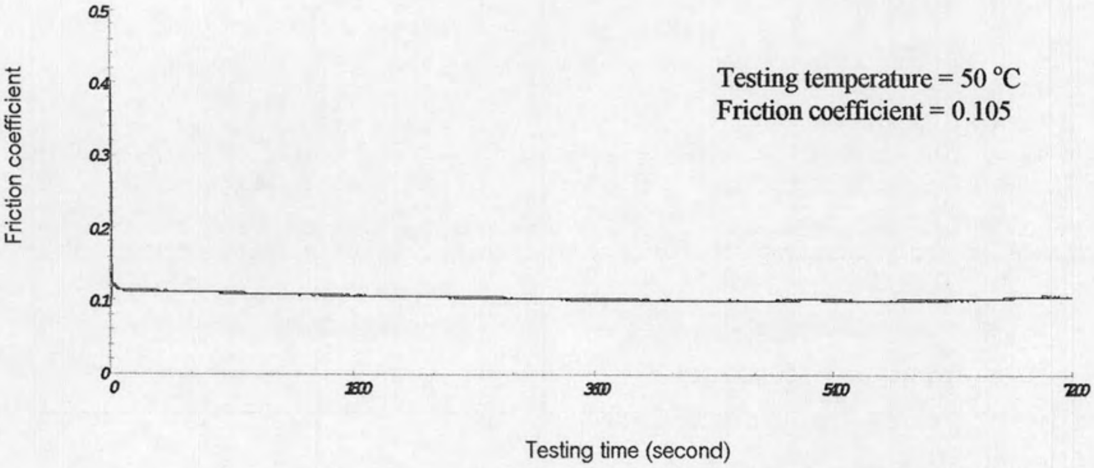


Figure B14 Graph of friction coefficient versus testing time at 50 °C of sample III

Sample VI

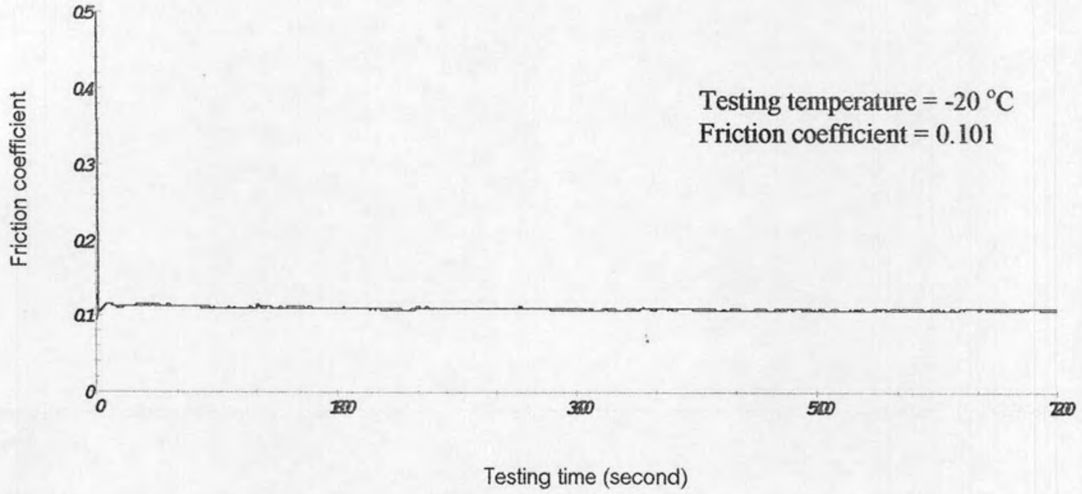


Figure B15 Graph of friction coefficient versus testing time at -20 °C of sample VI

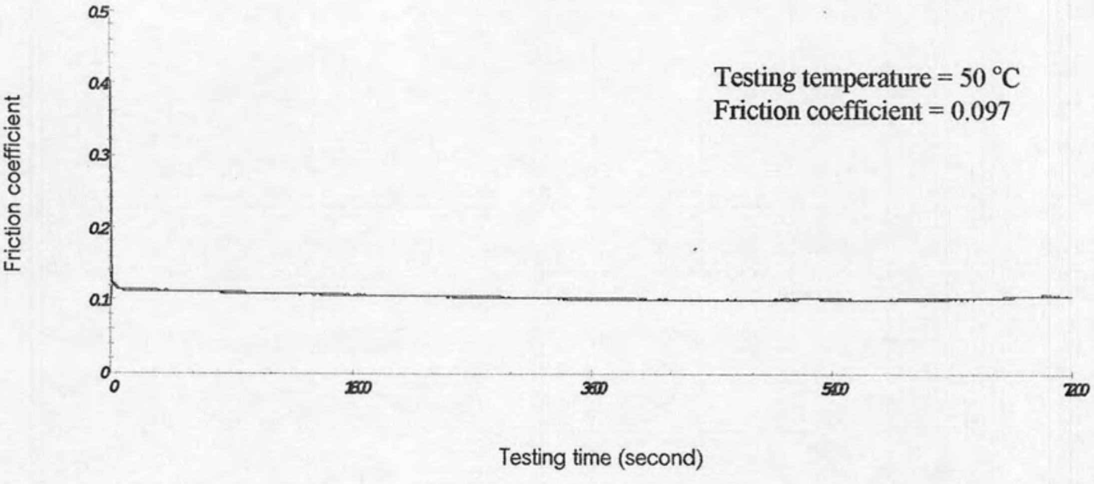


Figure B16 Graph of friction coefficient versus testing time at 50 °C of sample VI

Commercial B

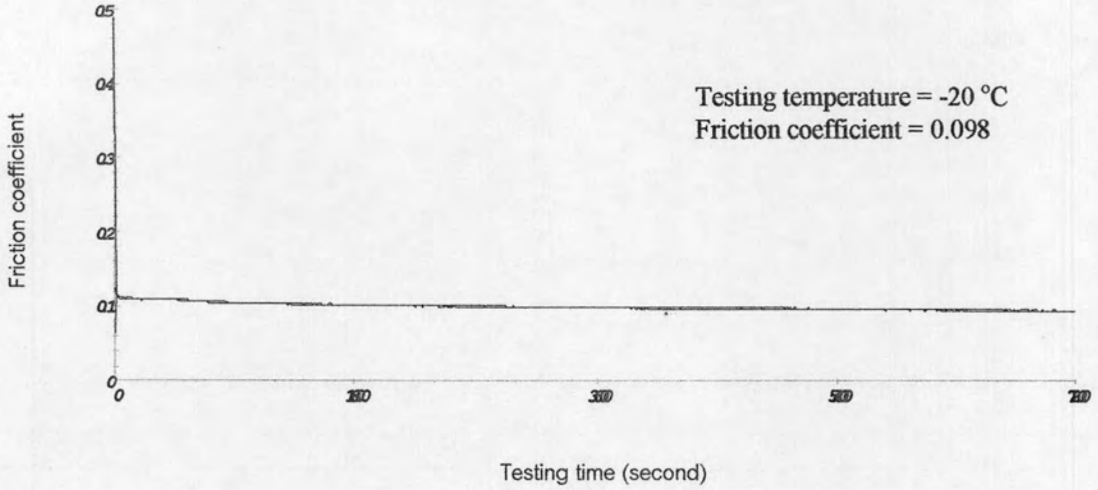


Figure B17 Graph of friction coefficient versus testing time at -20 °C of commercial B

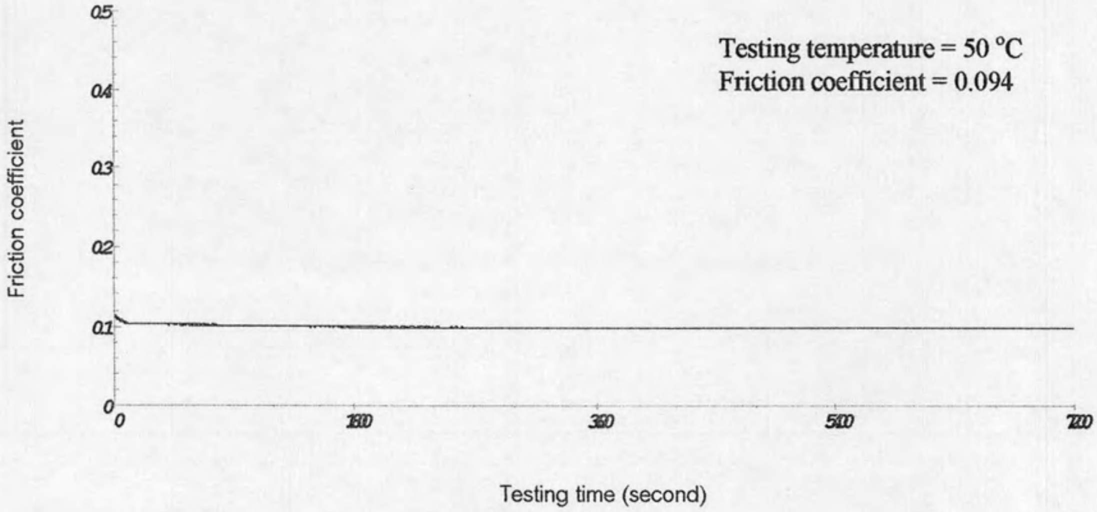


Figure B18 Graph of friction coefficient versus testing time at 50 °C of commercial B

APPENDIX C

FTIR SPECTRA

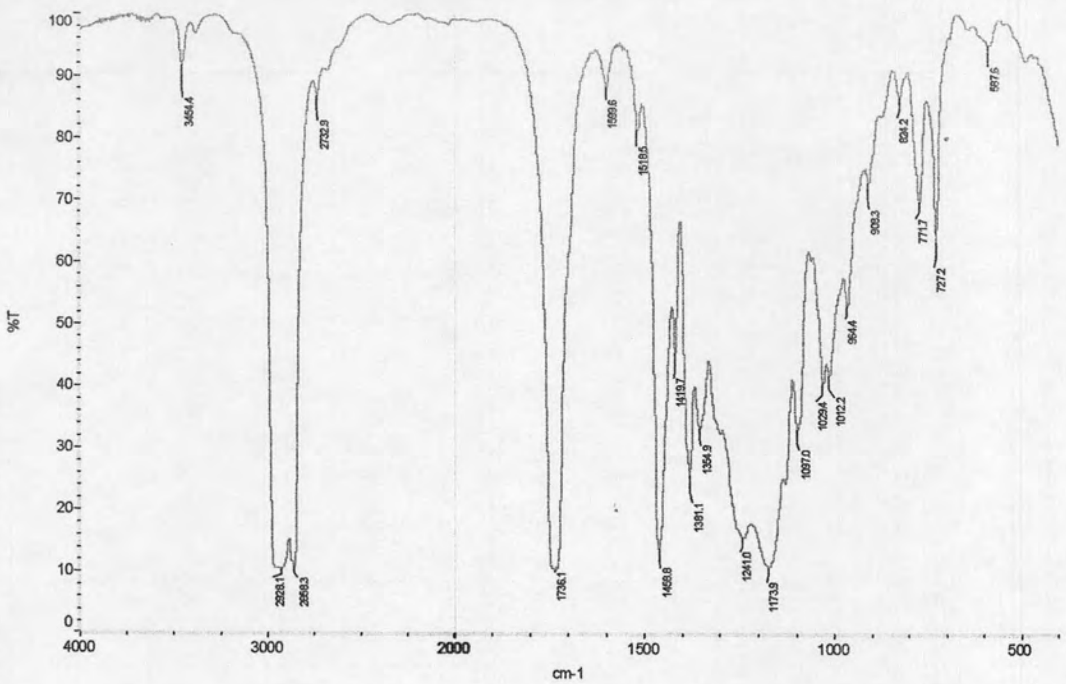


Figure C1 FTIR spectrum of sample II before compatibility test

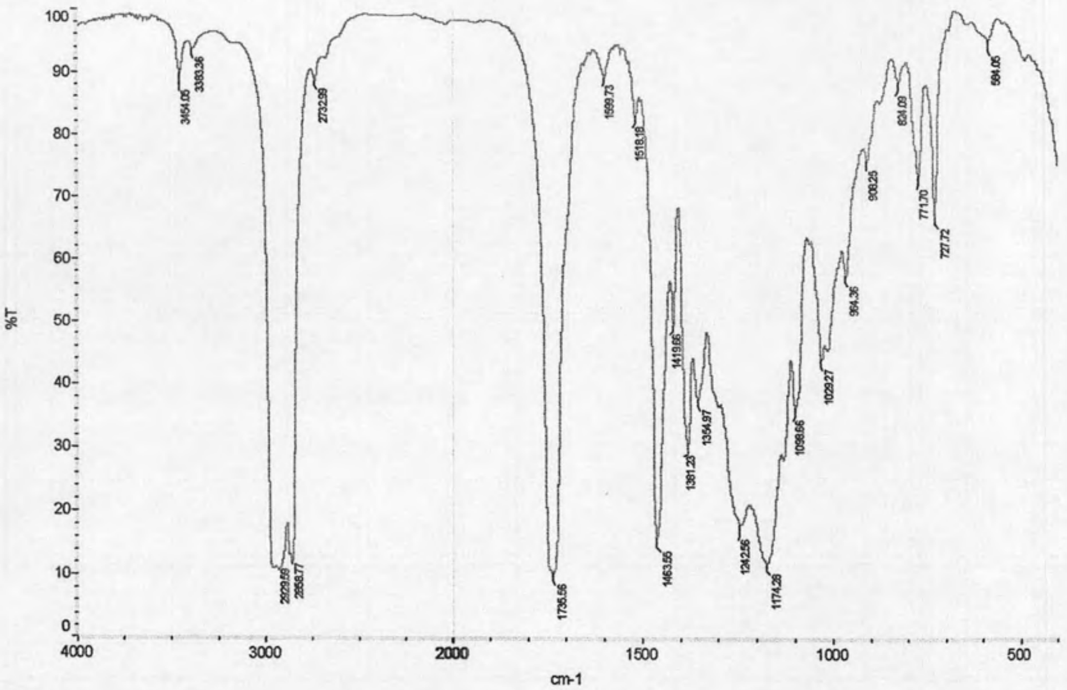


Figure C2 FTIR spectrum of sample II after compatibility test at 60 °C for 1 month

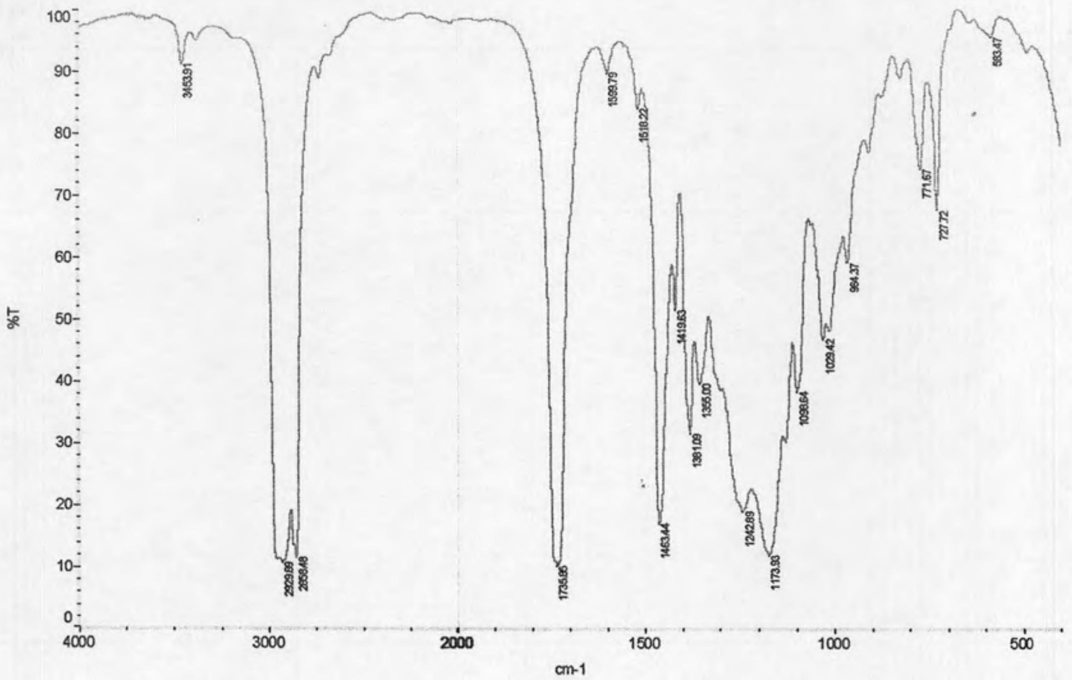


Figure C3 FTIR spectrum of sample II after compatibility test at 60 °C for 2 months

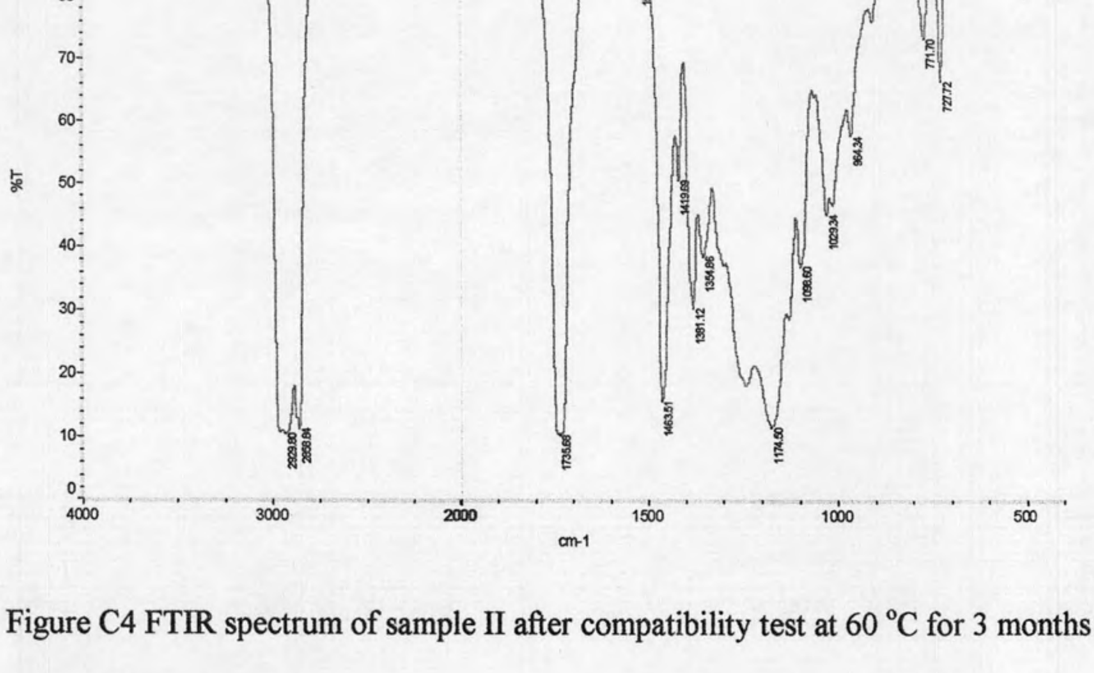


Figure C4 FTIR spectrum of sample II after compatibility test at 60 °C for 3 months

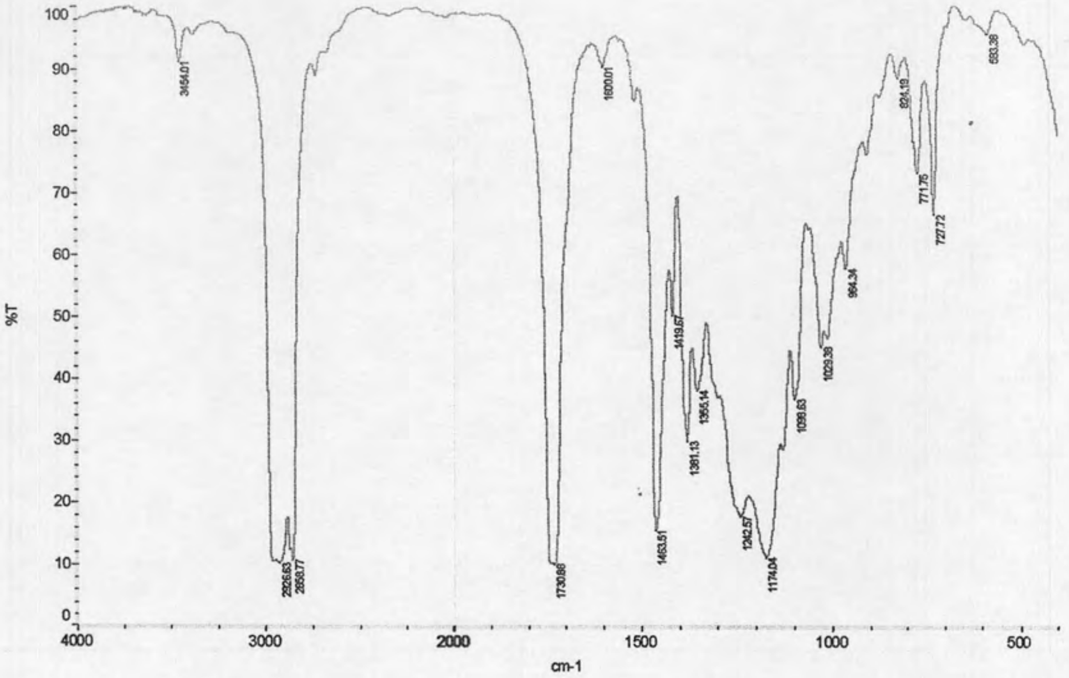


Figure C5 FTIR spectrum of sample II after compatibility test at 120 °C for 1 month

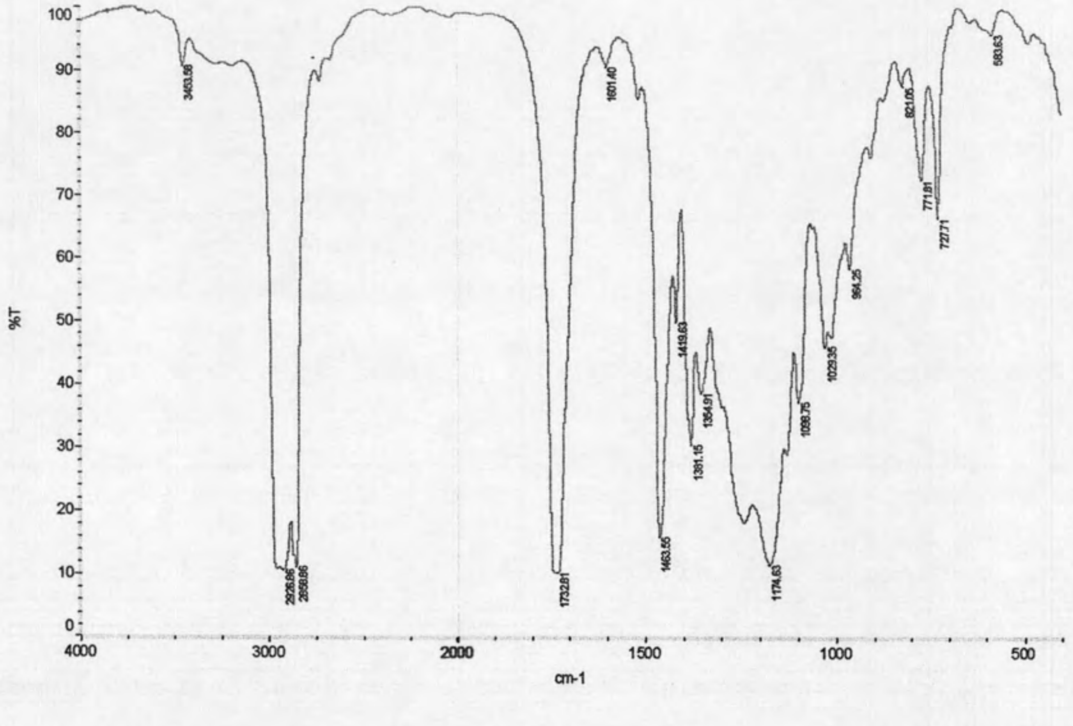


Figure C6 FTIR spectrum of sample II after compatibility test at 120 °C for 2 months

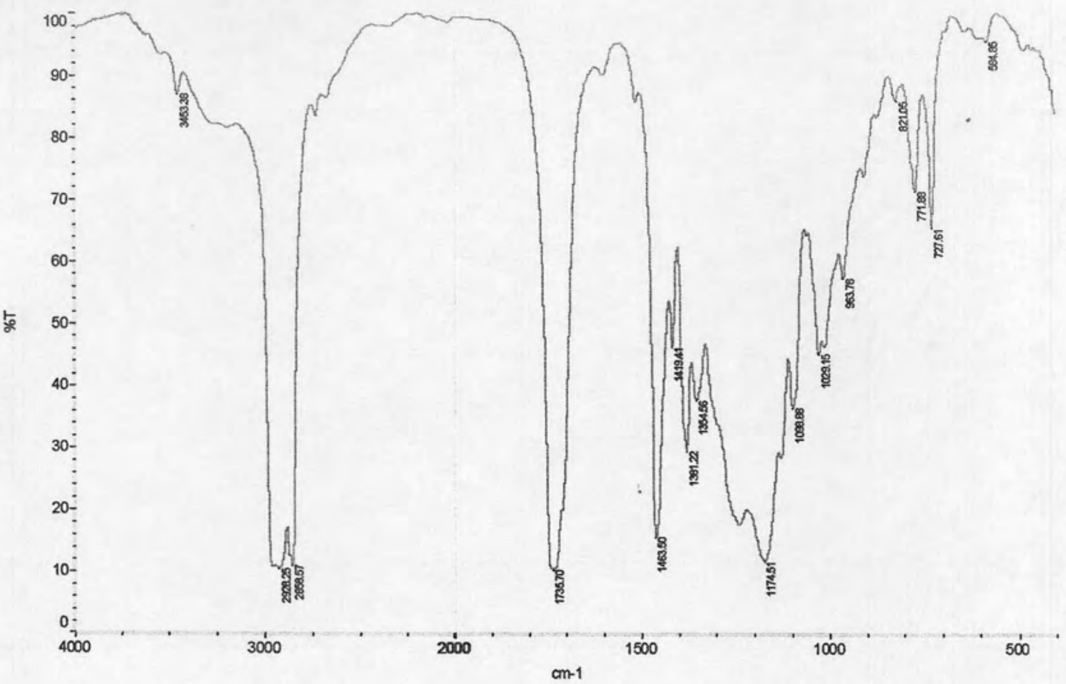


Figure C7 FTIR spectrum of sample II after compatibility test at 120 °C for 3 months

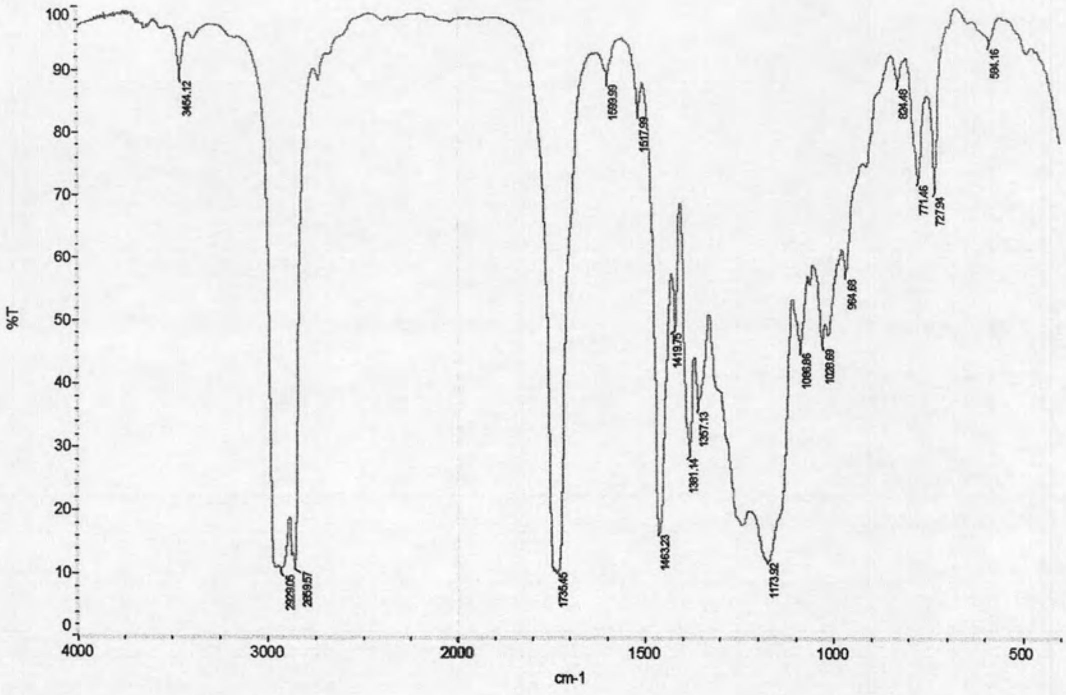


Figure C8 FTIR spectrum of sample III before compatibility test

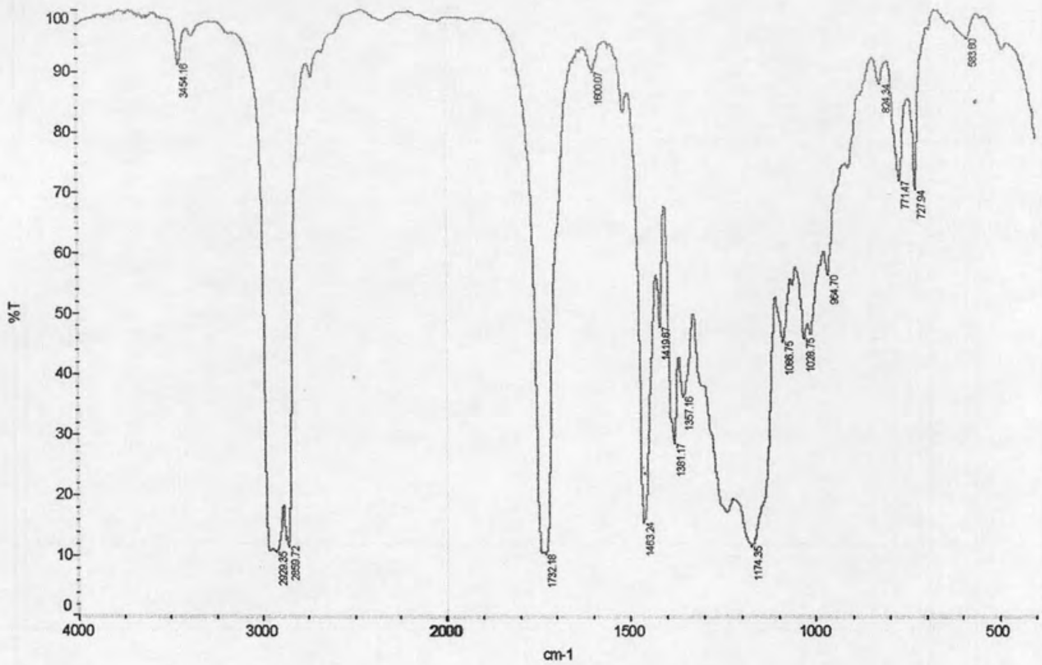


Figure C9 FTIR spectrum of sample III after compatibility test at 60 °C for 1 month

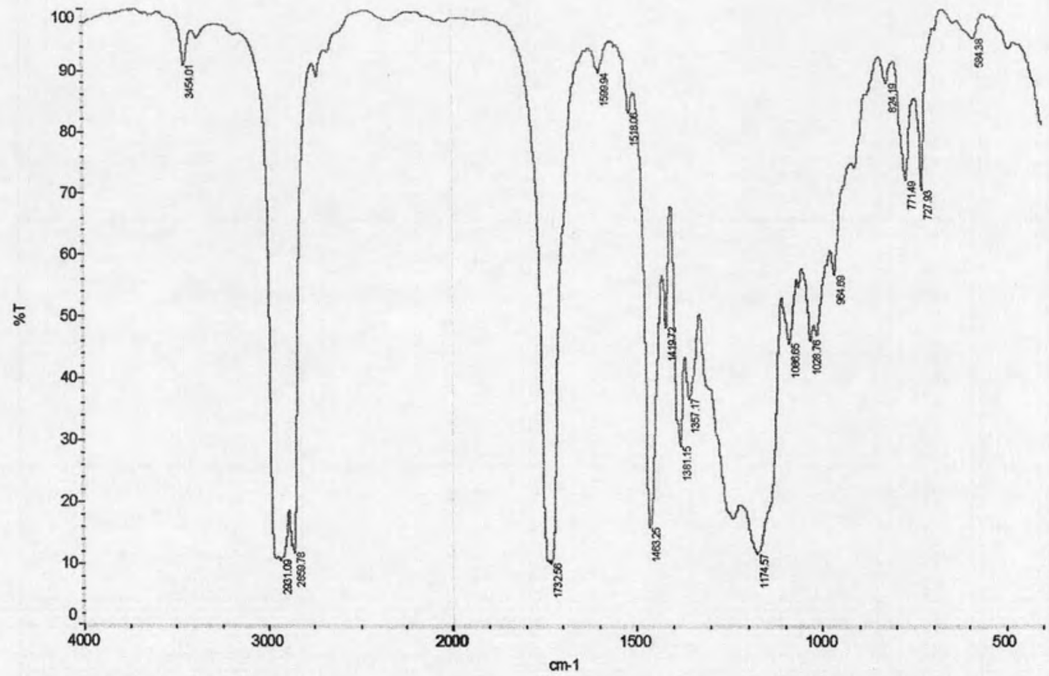


Figure C10 FTIR spectrum of sample III after compatibility test at 60 °C for 2 months

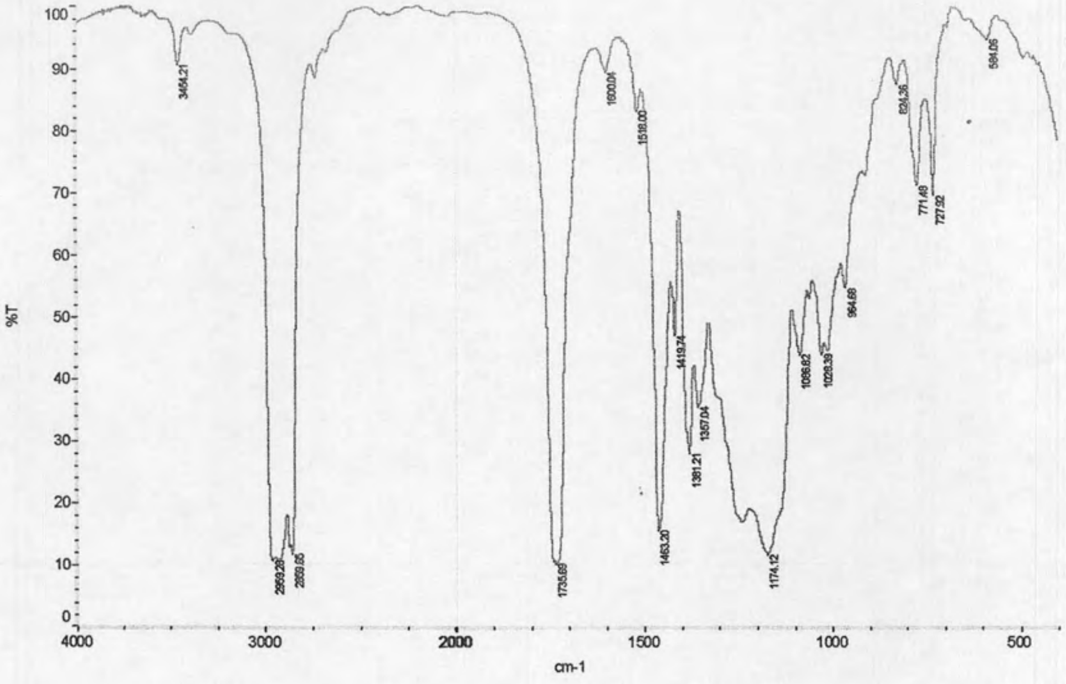


Figure C11 FTIR spectrum of sample III after compatibility test at 60 °C for 3 months

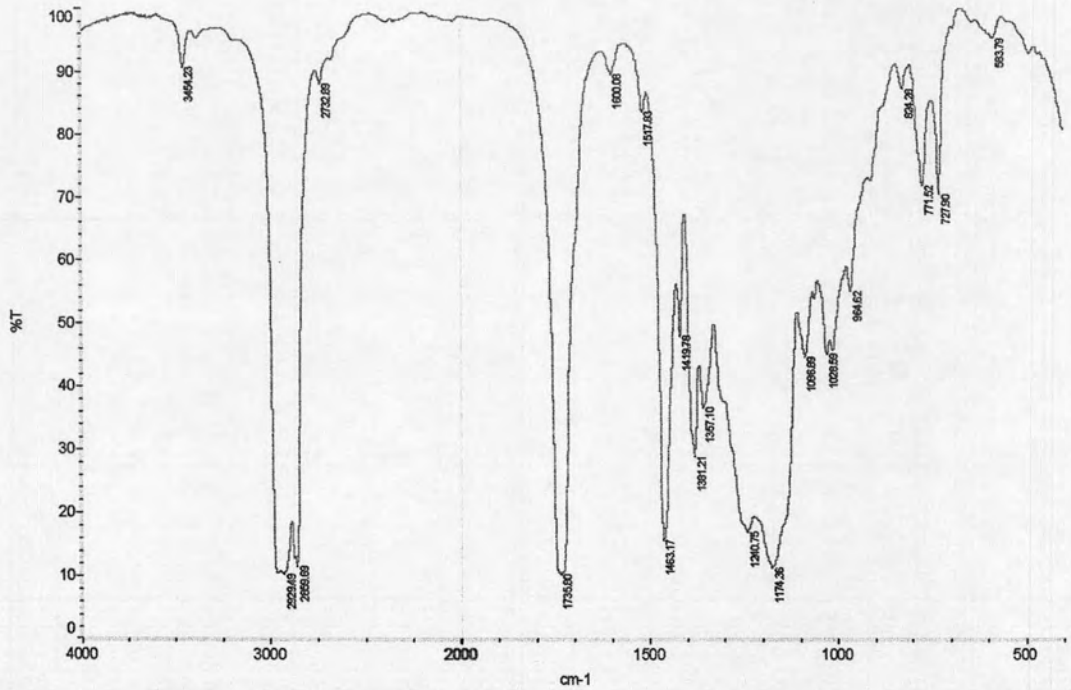


Figure C12 FTIR spectrum of sample III after compatibility test at 120 °C for 1 month

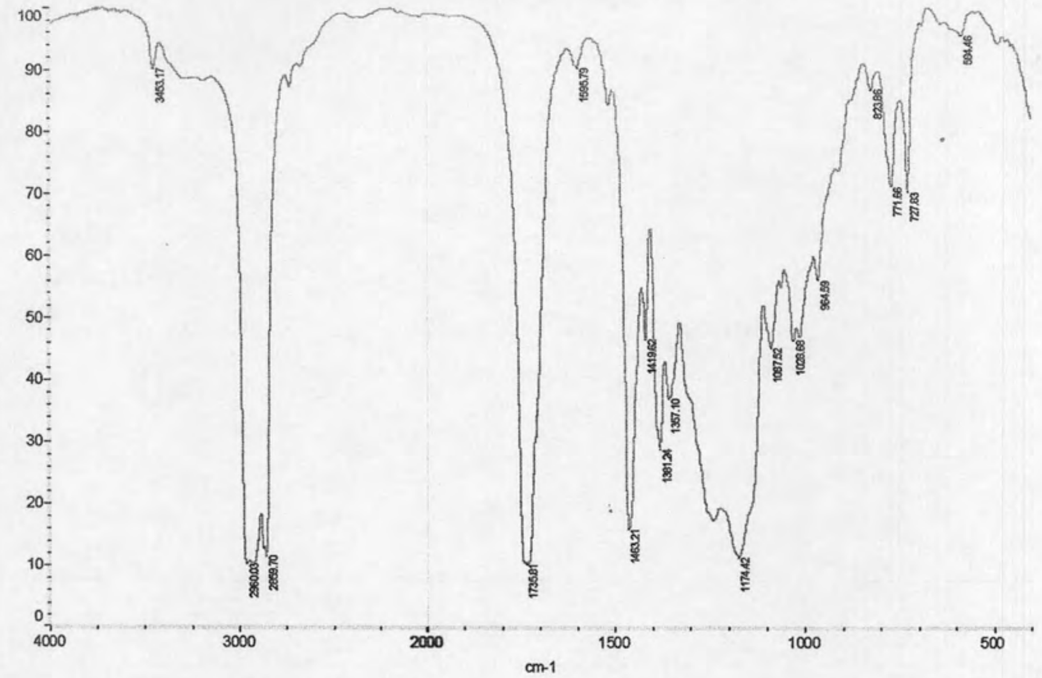


Figure C13 FTIR spectrum of sample III after compatibility test at 120 °C for 2 months

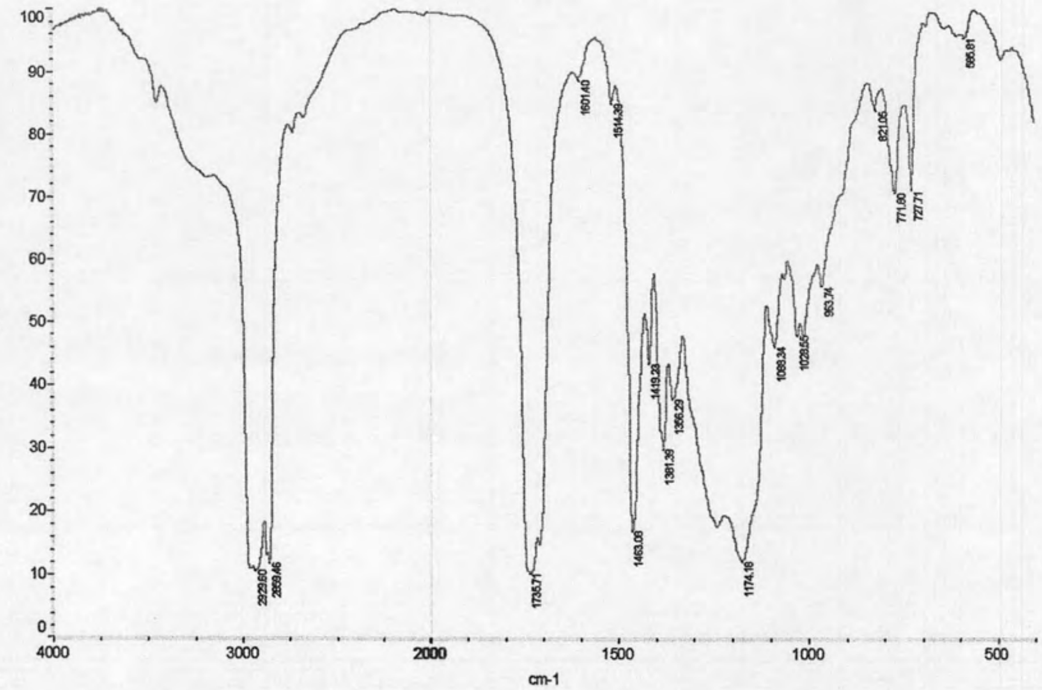


Figure C14 FTIR spectrum of sample III after compatibility test at 120 °C for 3 months

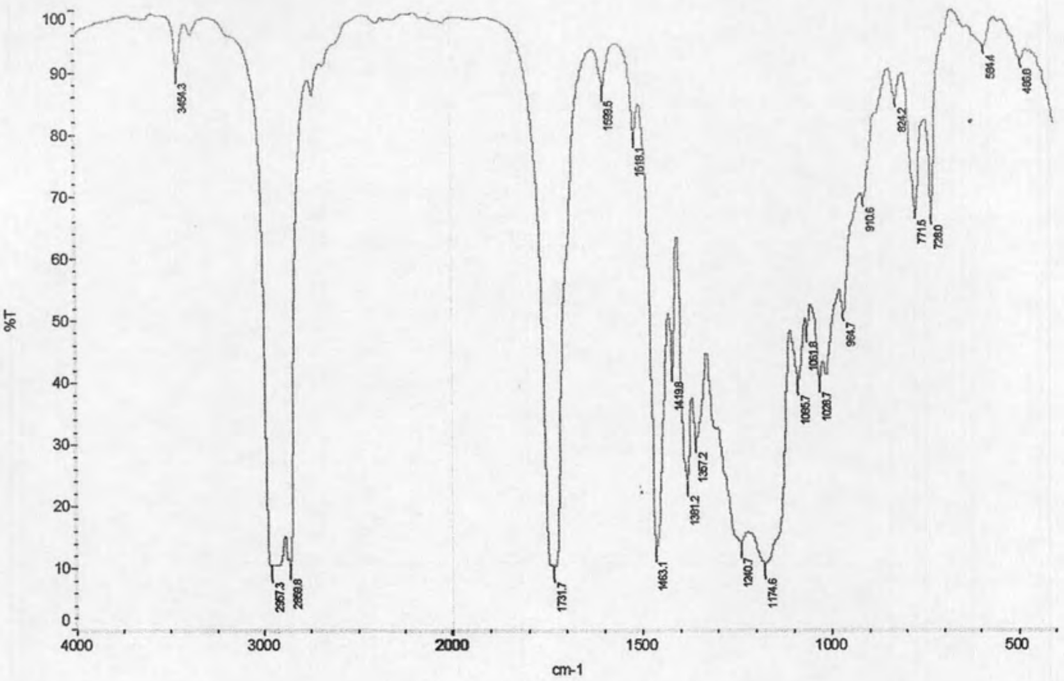


Figure C15 FTIR spectrum of sample VI before compatibility test

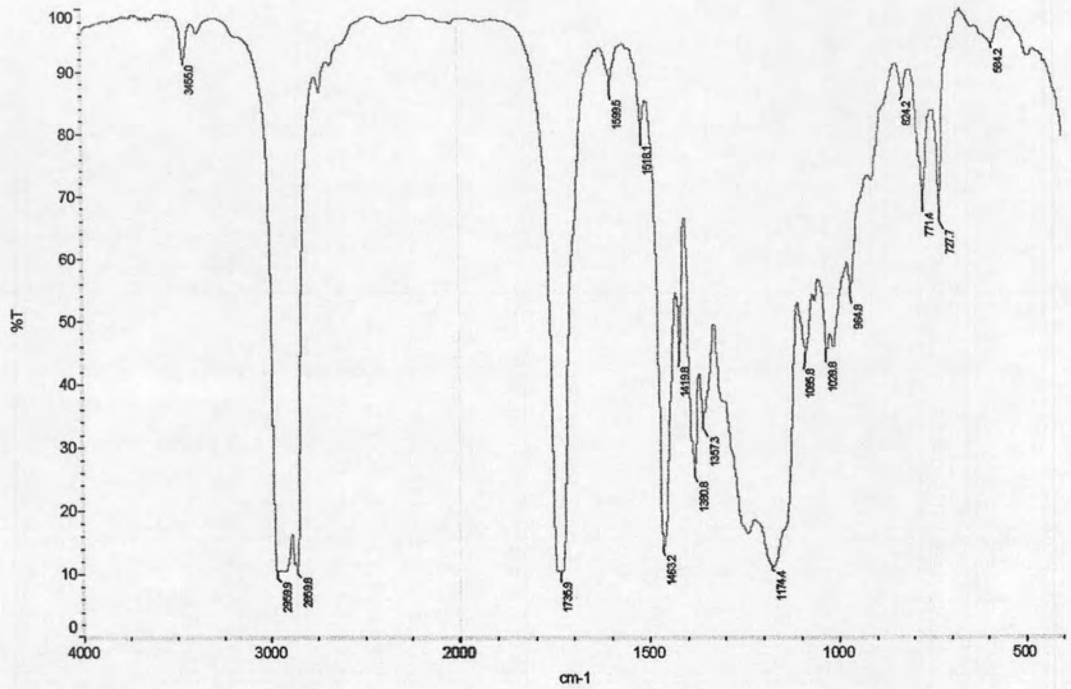


Figure C16 FTIR spectrum of sample VI after compatibility test at 60 °C for 1 month

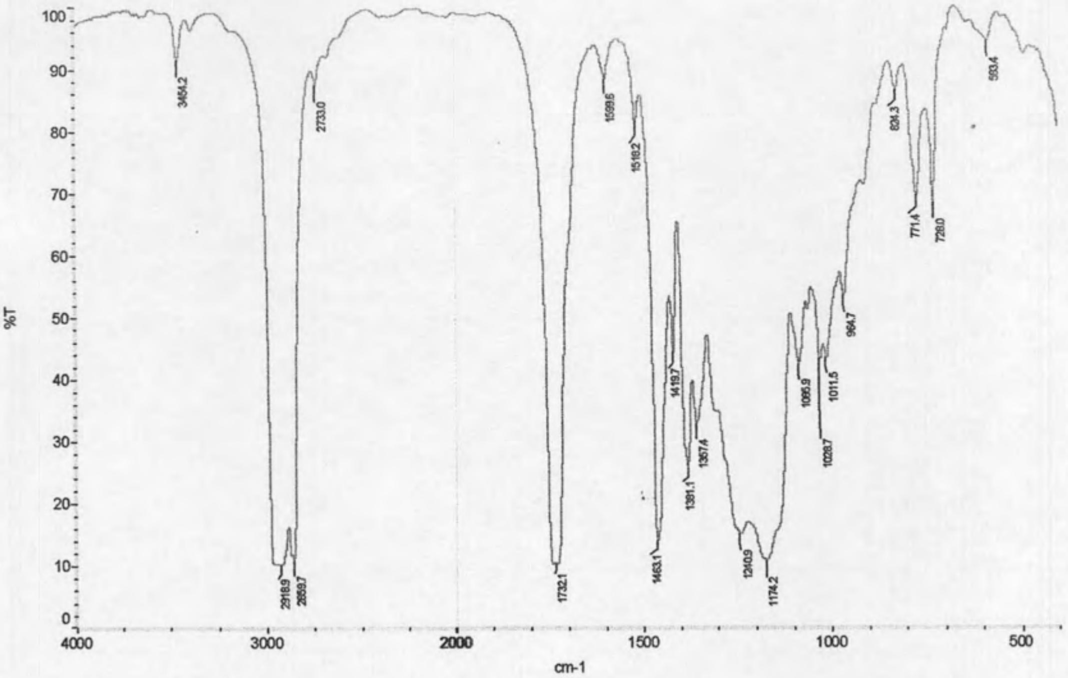


Figure C17 FTIR spectrum of sample VI after compatibility test at 60 °C for 2 months

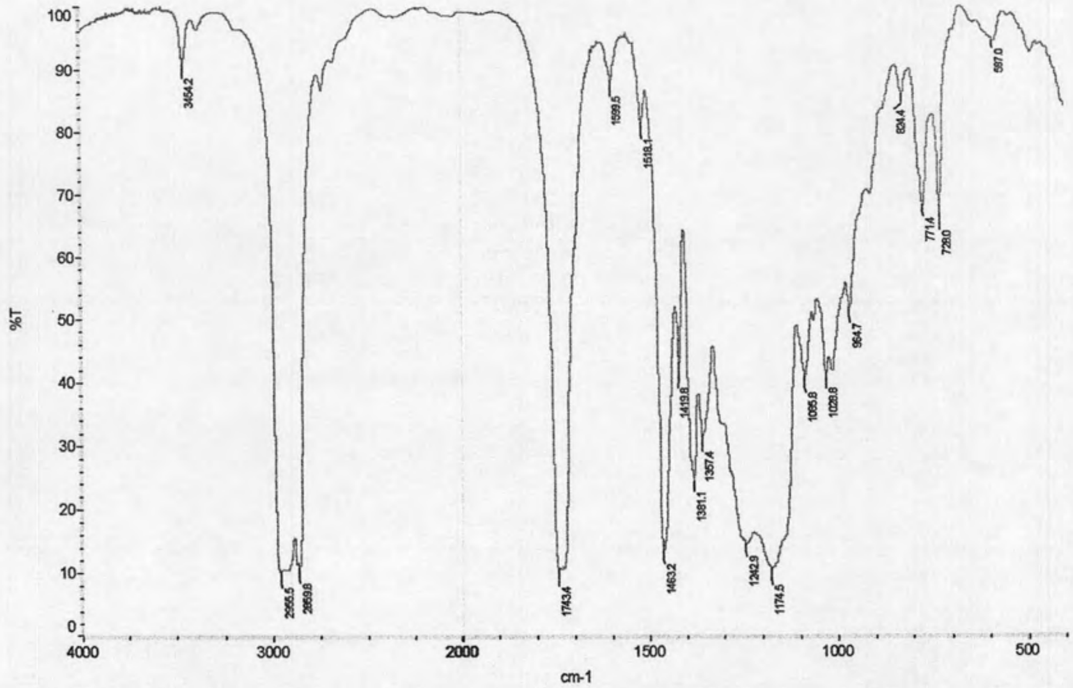


Figure C18 FTIR spectrum of sample VI after compatibility test at 60 °C for 3 months

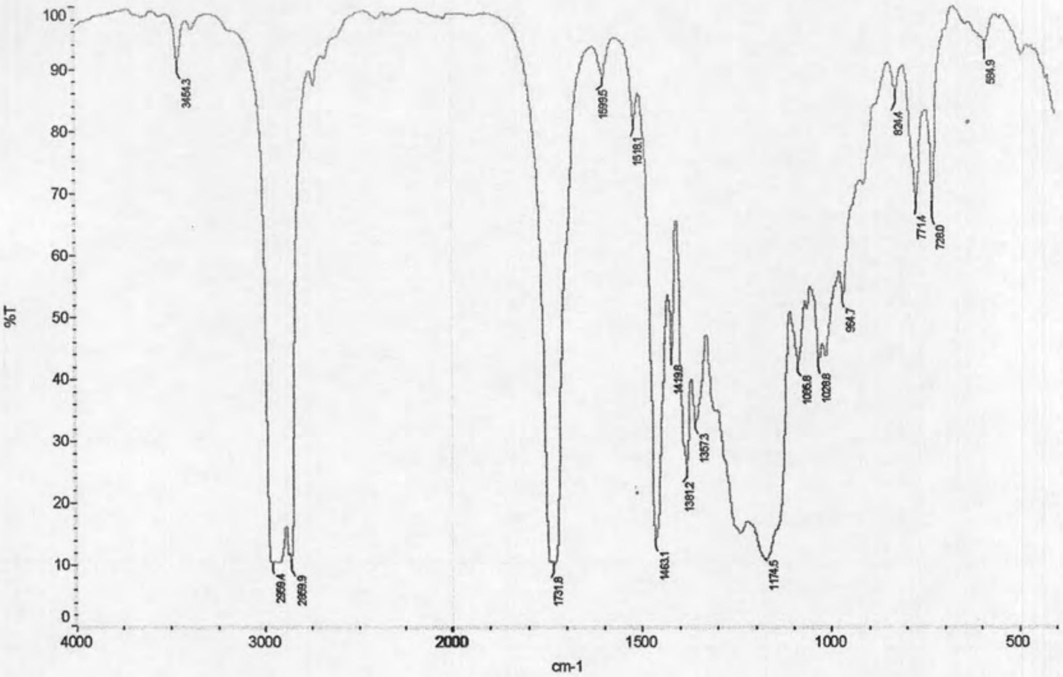


Figure C19 FTIR spectrum of sample VI after compatibility test at 120 °C for 1 month

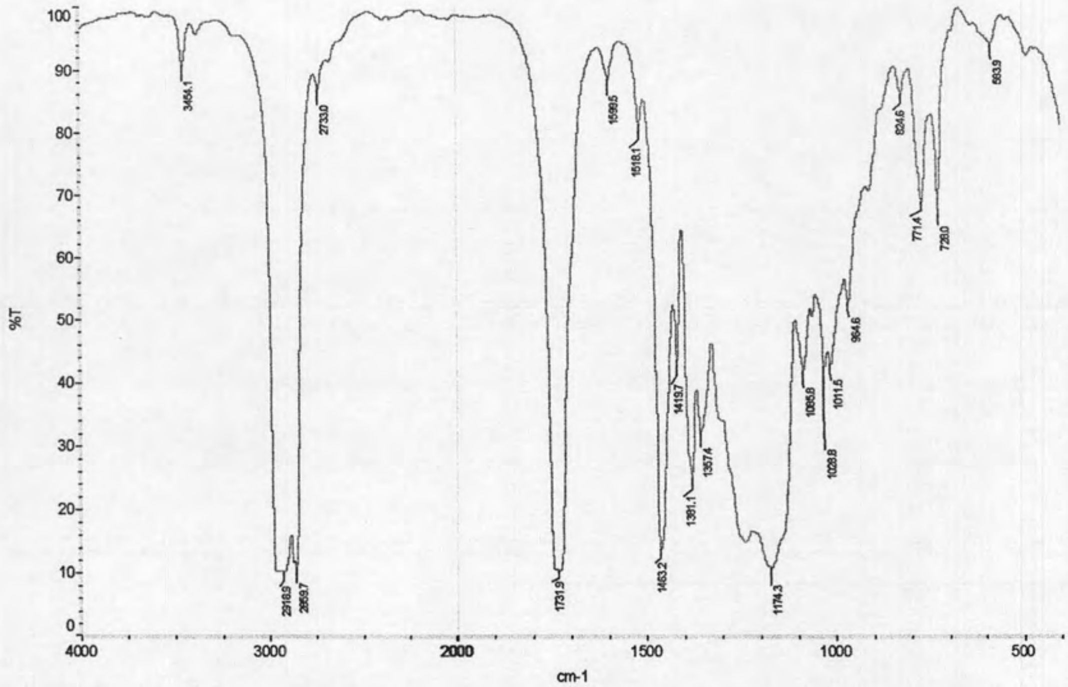


Figure C20 FTIR spectrum of sample VI after compatibility test at 120 °C for 2 months

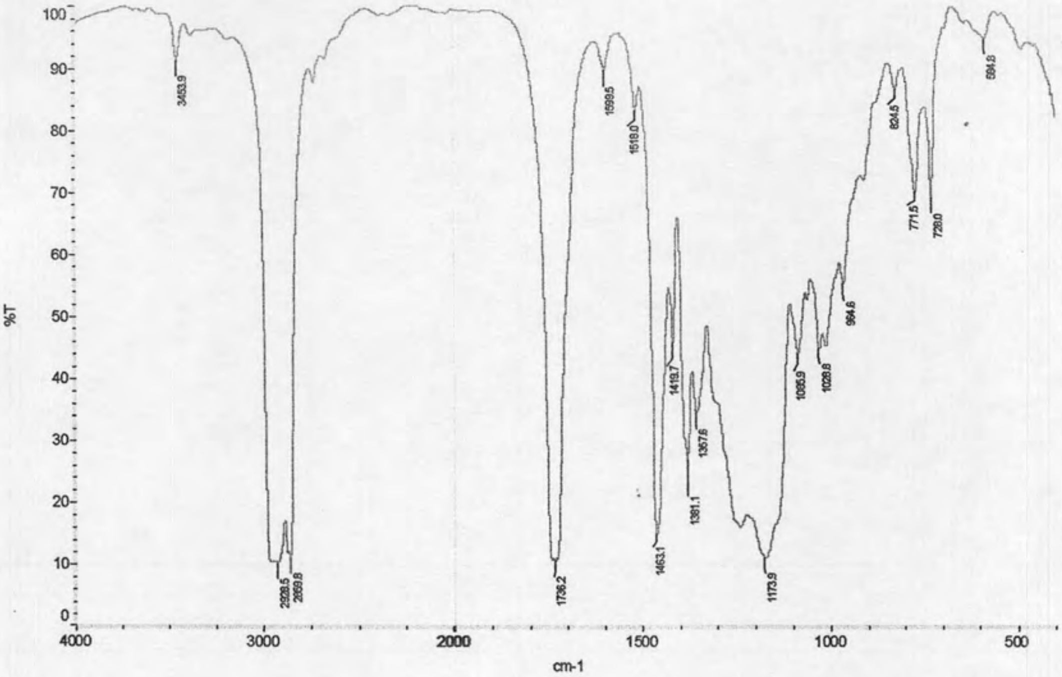


Figure C21 FTIR spectrum of sample VI after compatibility test at 120 °C for 3 months

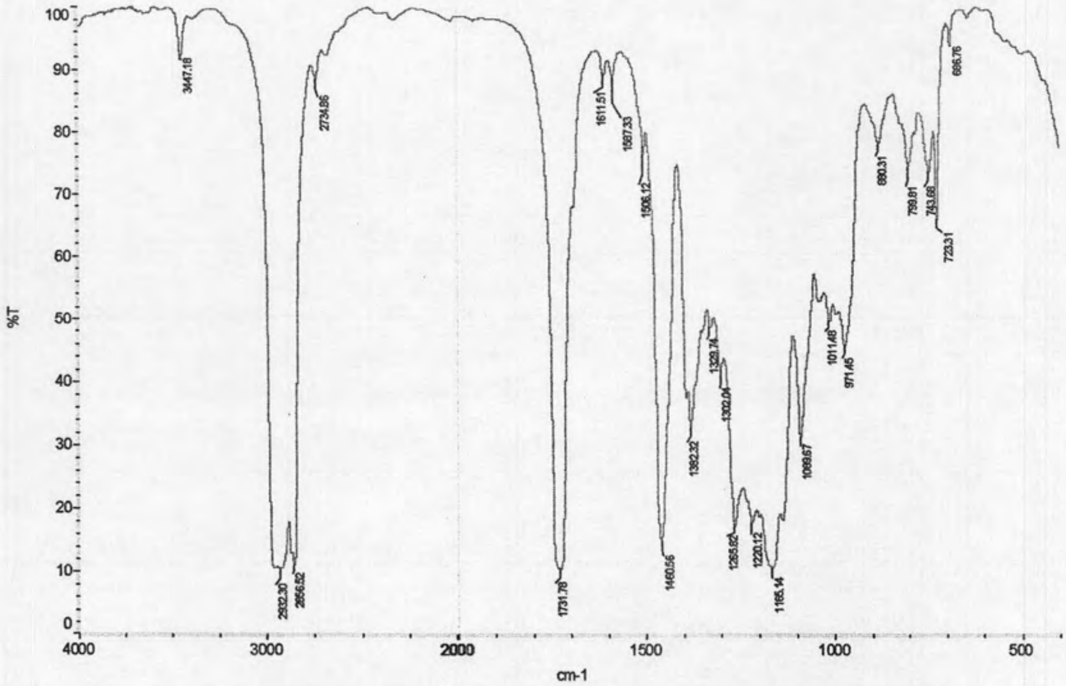


Figure C22 FTIR spectrum of commercial B before compatibility test

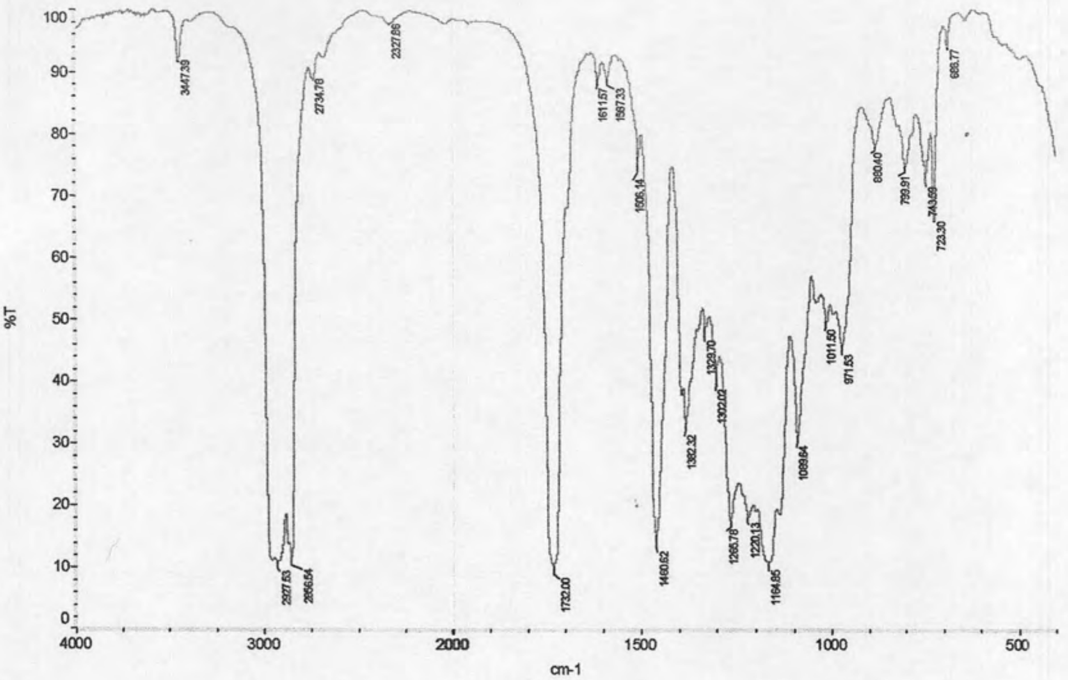


Figure C23 FTIR spectrum of commercial B after compatibility test at 60 °C for 1 month

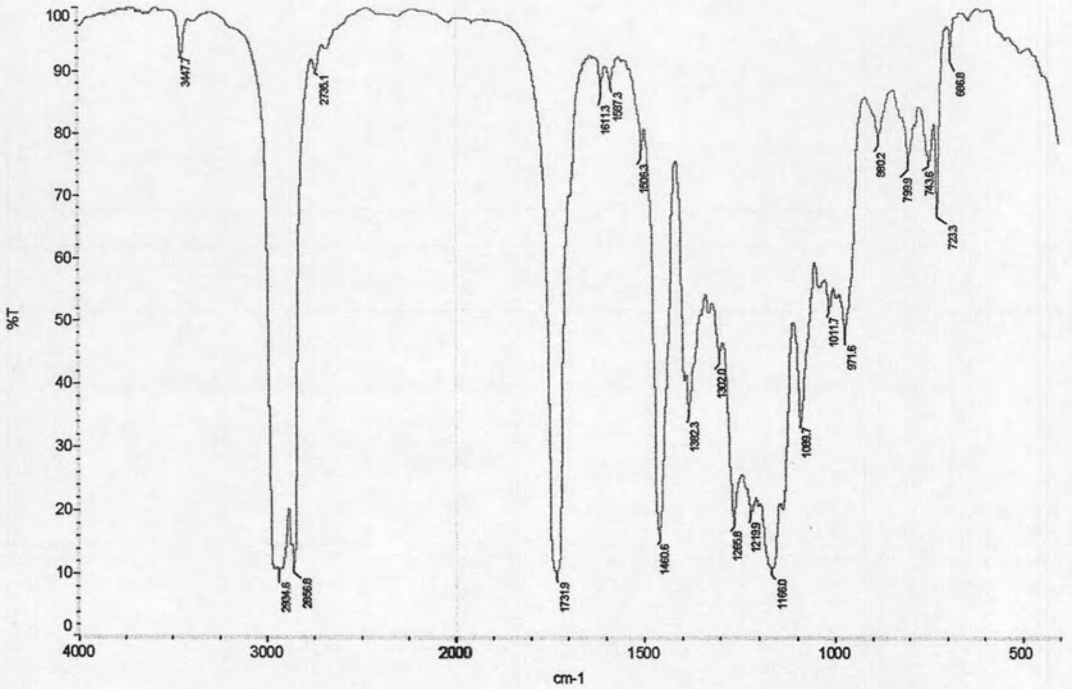


Figure C24 FTIR spectrum of commercial B after compatibility test at 60 °C for 2 months

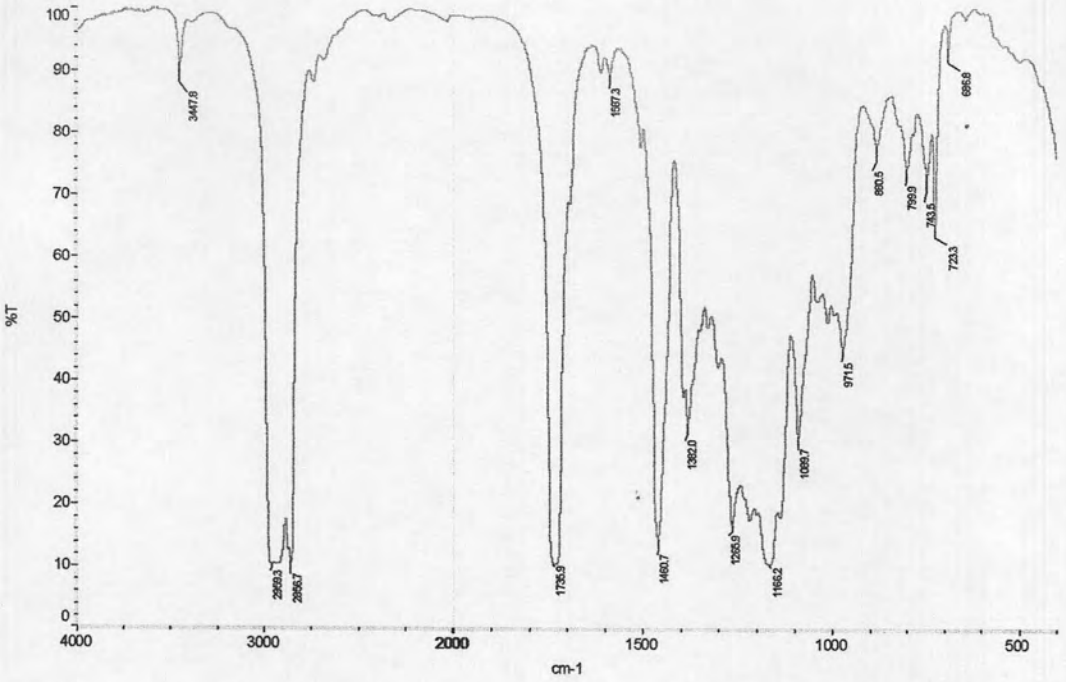


Figure C25 FTIR spectrum of commercial B after compatibility test at 60 °C for 3 months

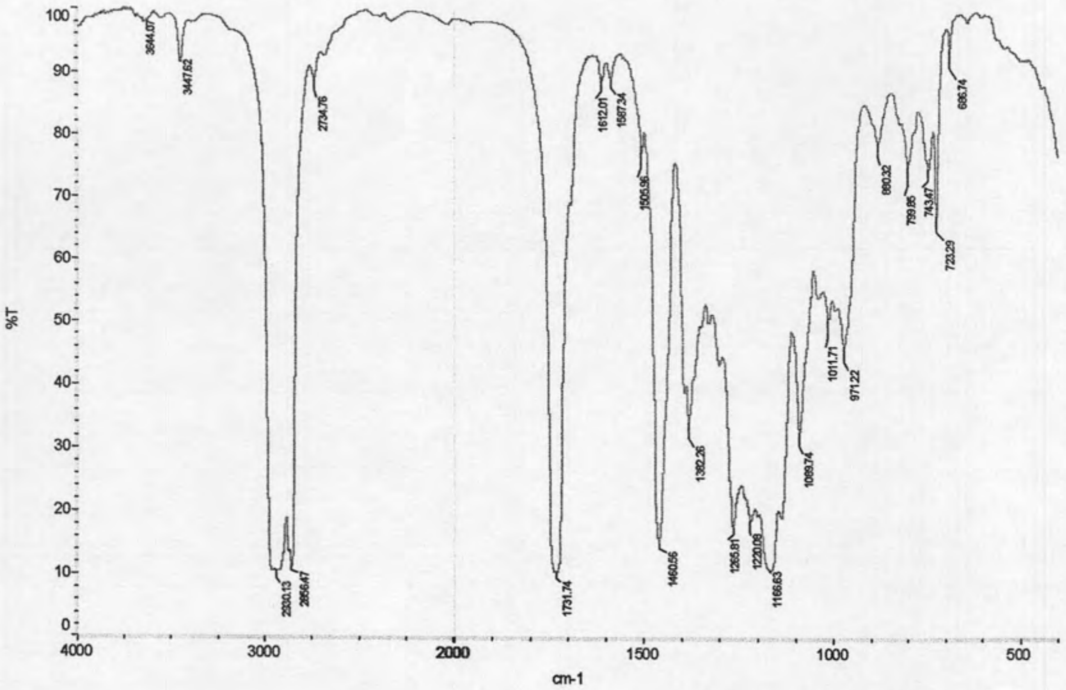


Figure C26 FTIR spectrum of commercial B after compatibility test at 120 °C for 1 month

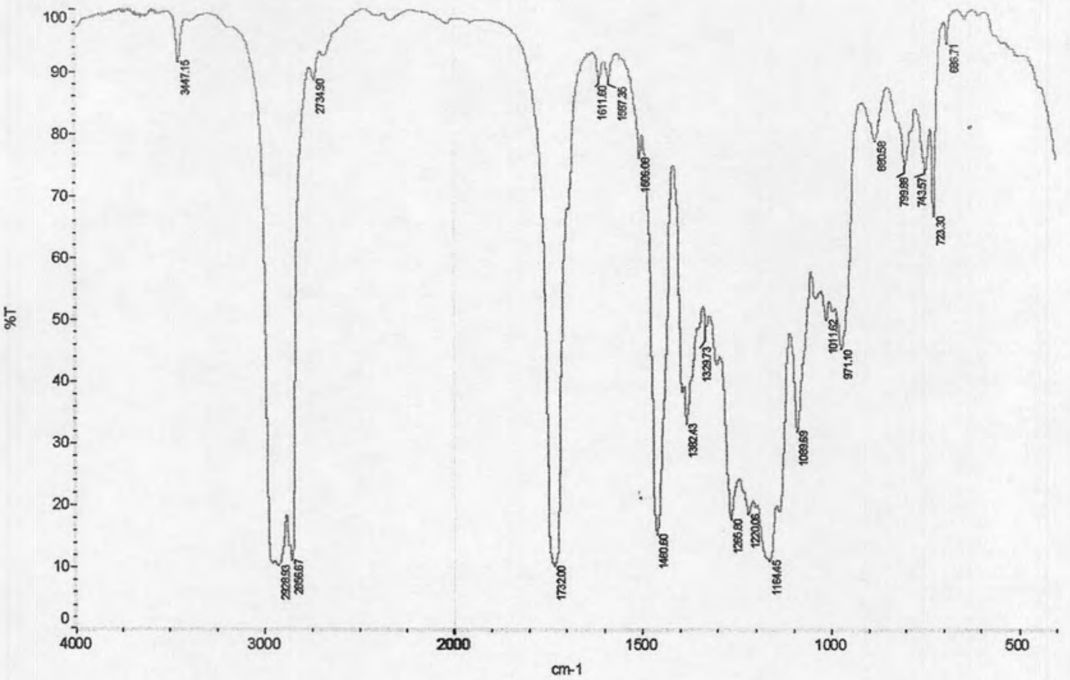


Figure C27 FTIR spectrum of commercial B after compatibility test at 120 °C for 2 months

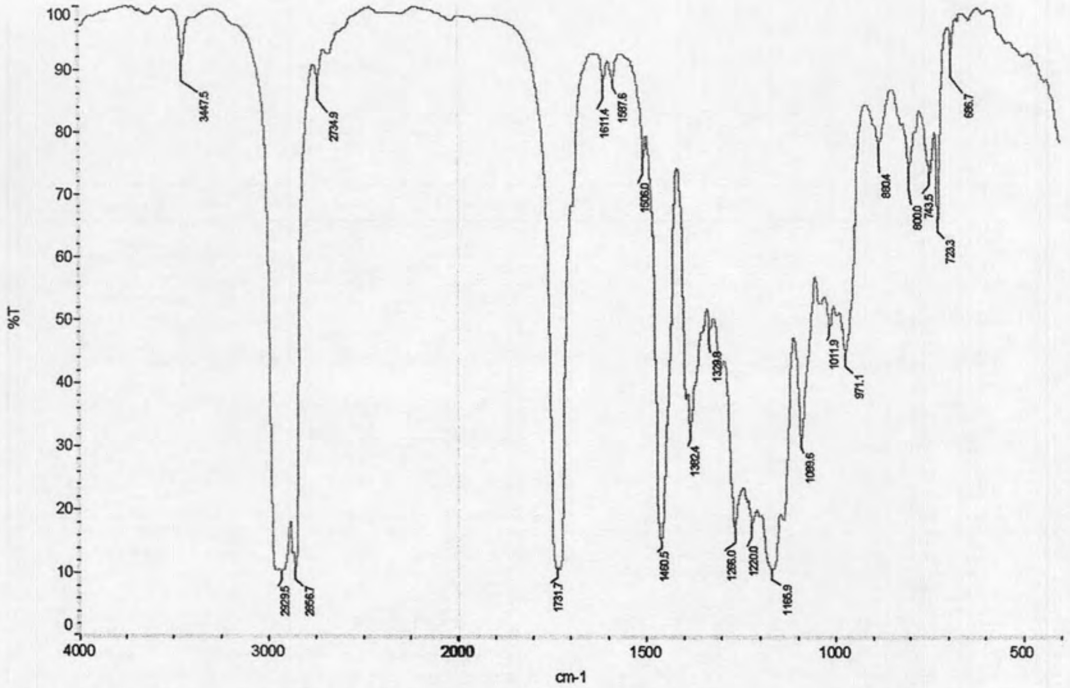


Figure C28 FTIR spectrum of commercial B after compatibility test at 120 °C for 3 months

APPENDIX D
MASS SPECTRA

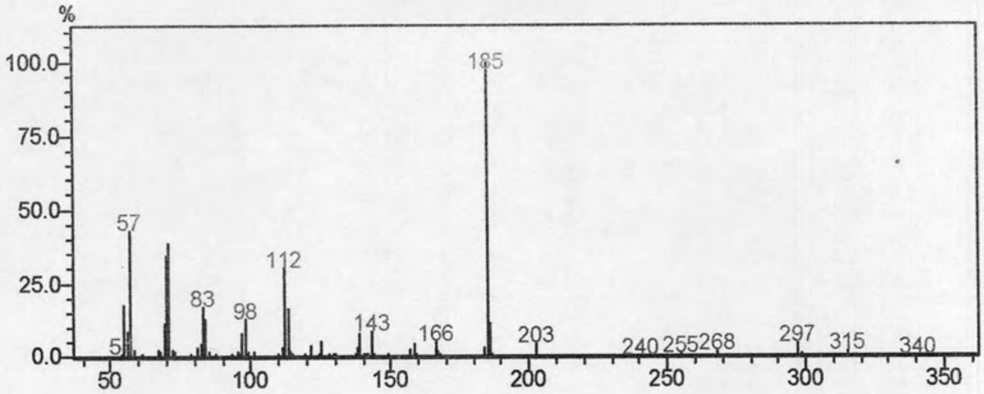


Figure D1 Mass spectrum of dioctyl sebacate

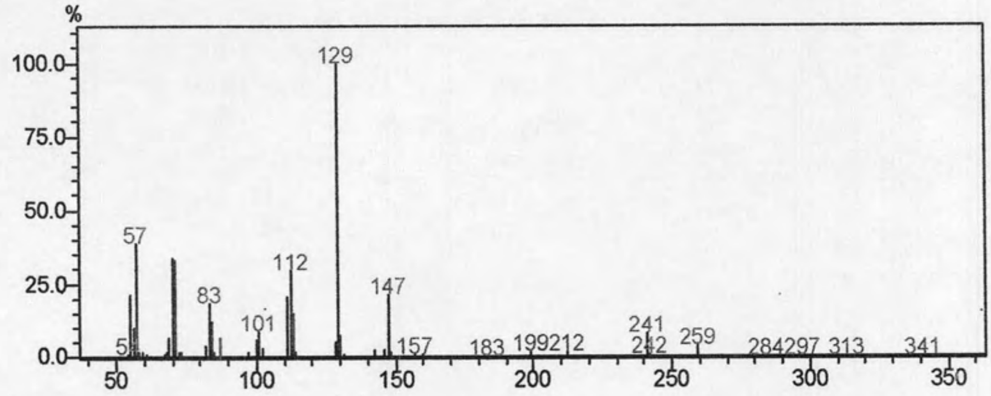


Figure D2 Mass spectrum of dioctyl adipate

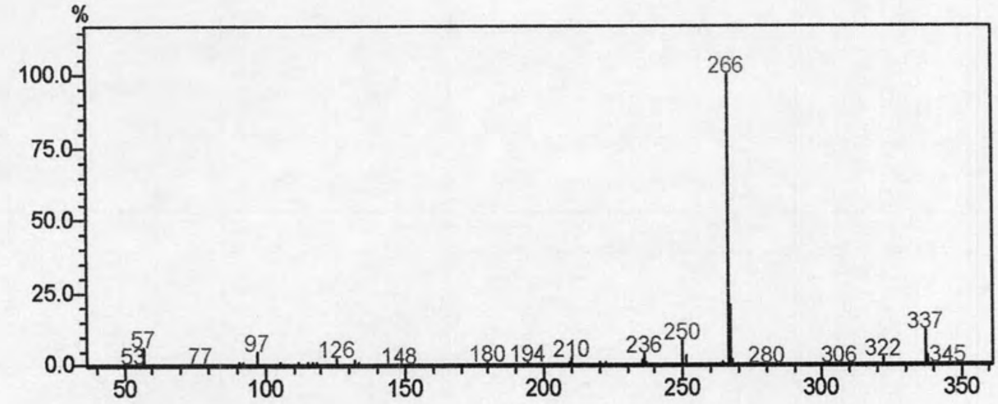


Figure D3 Mass spectrum of antioxidant

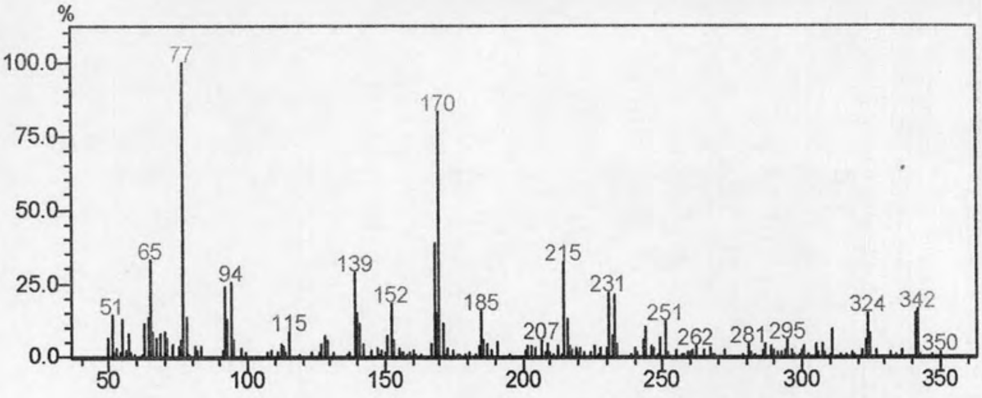


Figure D4 Mass spectrum of antiwear

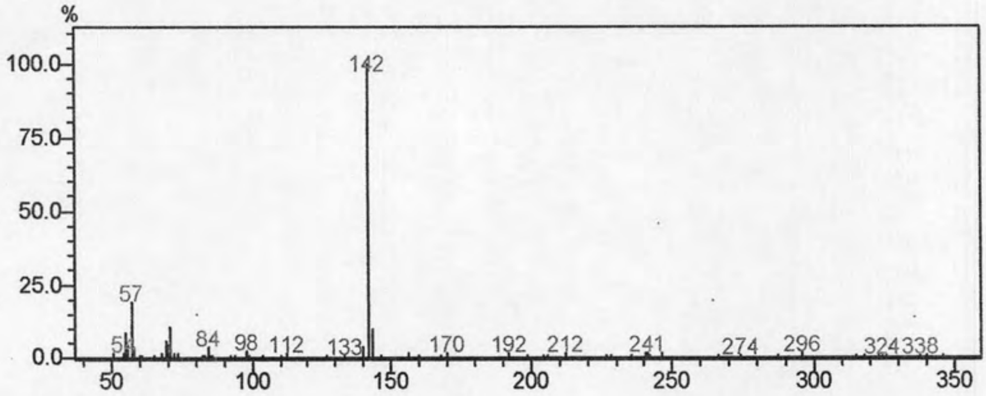


Figure D5 Mass spectrum of metal deactivator

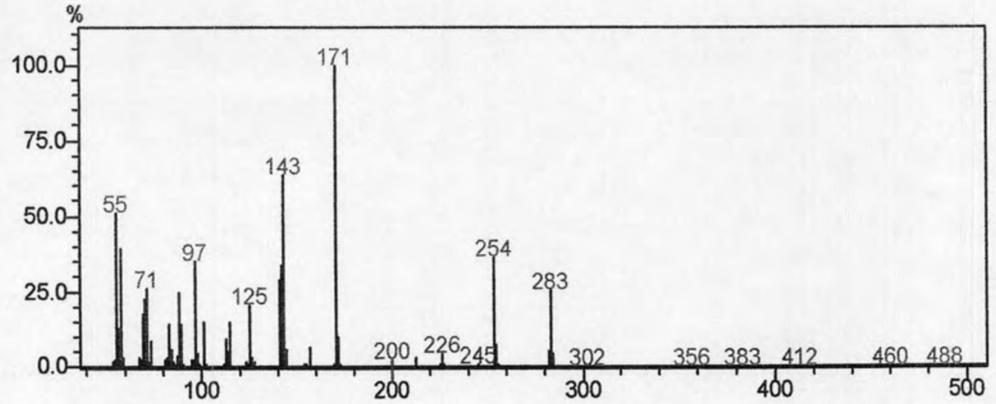


Figure D6 Mass spectrum of base oil of commercial B

VITA

Sumittra Duangkaewmanee was born on September 15, 1978, in Ayutthaya, Thailand, She received her Bachelor's degree of Science in Chemistry, Chulalongkorn University in 2001. She is researcher who works at R&D division, Minebea Ltd.,Thailand. She attended the Master's Degree of Science Program in Petrochemistry and Polymer Scince, Faculty of Science, Chulalongkorn University. She completed the program in 2006.