

CHAPTER III

EXPERIMENTAL

3.1 Chemicals

Lithium hydroxide monohydrate ($\text{LiOH}\cdot\text{H}_2\text{O}$, melting point $470\text{ }^\circ\text{C}$) and lithium stearate ($\text{C}_{17}\text{H}_{35}\text{COOLi}$, melting point $205\text{ }^\circ\text{C}$) were purchased from Carlo Elba Company. Stearic acid ($\text{C}_{17}\text{H}_{35}\text{COOH}$, melting point $70\text{ }^\circ\text{C}$) and paraffin oil were purchased from Vidhyasom Co., Ltd. Trimethylol propane ester (TMP-ester), lithium-ester grease and lithium stearate were obtained from Nippon Grease Company.

Zinc dialkyl dithiophosphate (ZDDP) and molybdenum dithiocarbamate (MDTC) were obtained from Lubrizol Company. Diphenylamine (DPA) was obtained from Vanalube Company. Hydranol-solvent CM (a mixture of trichloromethane and methanol) and hydranol composite-5 (a mixture of imidazole, sulfur dioxide and iodine) were obtained from Riedel-deHaen Company.

3.2 Instrument and apparatus

- 3.2.1 Cannon-Fenske viscometer, model series 200 from RIGOSHA Co., Ltd.
- 3.2.2 Cleveland open cup test unit, model ACO-5 from TANAKA.
- 3.2.3 Automatic pour point tester, model RPP-02 CML from RIGOSHA Co., Ltd.
- 3.2.4 Micro melting point apparatus model MPP-100 from YAHACO
- 3.2.5 Karl-Fisher moisture titrator with moisture evaporator, model MKA-210 from Kyoto Electronics Co., Ltd.
- 3.2.6 A three-roll mill machine, model MB-10V from Inoue Seisakusho.

- 3.2.7 Apparatus for penetration and work stability test are a half-scale penetrometer with a brass cone, a motor grease worker machine and a grease workers with 1/4 inch holes.
- 3.2.8 Apparatus for dropping point are a grease cup, a special test tube, thermometers, a 400-ml beaker with silicone oil and a metal rod (12 cm. in length and 1.2 mm. in diameter).
- 3.2.9 Apparatus for copper corrosion are test tubes, a copper strip (75 mm in length, 12.5 mm. in width and 3 mm. thickness) and silicon carbide abrasive paper (240-grit and 150-mesh) were used.
- 3.2.10 Evaporation cell with air supply system, model 848-03 from RIGOSHA Co., Ltd.
- 3.2.11 Apparatus for oil separation are a 60-mesh screen cones with hanger and a 100 ml beaker with lid.
- 3.2.12 Norma-Hoffman oxidation bomb, model 854-03 from RIGOSHA Co., Ltd.
- 3.2.13 Apparatus for corrosion preventive properties are a bearing from MINEBEA Ltd., a glass jar and a microscope model BX-50 from OLYMPUS.
- 3.2.14 Gas chromatograph-massspectrometer (GC-MS), model HP 5890/5972 from Hewlett Packard and a automatic thermal desorption system (ATD) from Perkin Elmer.
- 3.2.15 Scanning electron microscope (SEM), model JSM 6330F from JEOL.
- 3.2.16 Fourier transform infrared spectrometer (FT-IR), model Paragon 1000 from Perkin Elmer.
- 3.2.17 X-ray fluorescence spectrometer, model RIX 3000 from RIGAKU.

3.3 Procedure

3.3.1 Preparation of lithium stearate

Lithium hydroxide monohydrate (0.22 kg) was dissolved in deionized water (200 ml) in a stainless steel pot at room temperature. Then the pot was placed on a hot plate at 60 °C. Stearic acid (1.47 kg) was put into another stainless steel pot, and heated on the hot plate at 90 °C. The lithium hydroxide solution was slowly poured into melted stearic acid, and the mixture was heated at 90 °C and stirred in the same time. After stirring for 20 minutes, the water was evaporated on the hot plate. The remaining of moisture was removed from the prepared lithium stearate using a vacuum oven at 60 °C for 4 hours. Lithium stearate (1.5 kg) as a white powder was obtained.

3.3.2 Preparation of lithium grease

3.3.2.1 Base grease preparation

The base oil, TMP ester oil or paraffin oil, and lithium stearate were mixed in a stainless steel pot. The ratio of base oil and lithium stearate used is shown in Table 3.1.

Table 3.1 Ratio of base oil and lithium stearate for grease preparation

Ratio of base oil :	80:20	82:18	85:15	90:10
Lithium stearate				
Base oil (g)	240	246	255	270
Lithium stearate (g)	60	54	45	30

The mixture was heated at 220 °C until a clear solution was obtained. The solution was poured into a stainless steel tray, and then cooled to the room temperature.

The base grease was homogenized by the three-roll mill machine (Figure 3.1) at pressure 2 kgf/cm² which distance between two beside roll and a center roll was set at 0.5 mm.

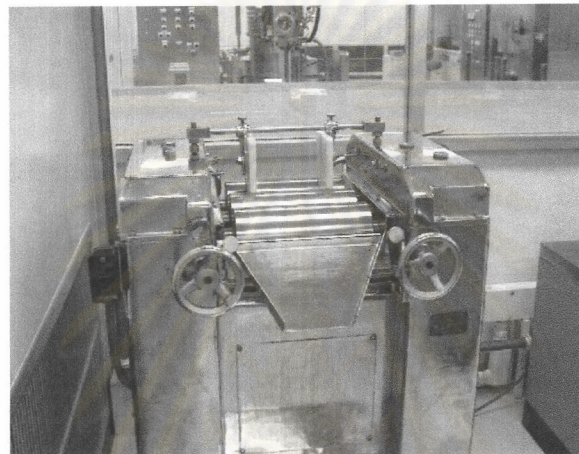


Figure 3.1 Three-roll mill machine for homogenized grease.

3.3.2.2 Finished grease preparation

TMP ester oil (2.46 kg) and paraffin oil (2.55 kg) were mixed with lithium stearate 0.54 kg and 0.45 kg, respectively. Each set of the mixture was mixed in a stainless steel pot and heated at 220 °C until both become a clear solution. The solution was poured in a stainless steel tray to cool down at the room temperature.

The appropriate amounts of additives were added into base grease as shown in Table 3.2. The mixture was blended at speed 20 rpm for 20 minutes. The grease was homogenized by the three-roll mill machine at pressure 2 kgf/cm².

Table 3.2 Weight of base grease and additive

	ZDDP			MDTC			DPA		
Amount of additive (g)	10	20	30	10	20	30	10	20	30
Amount of base grease (g)	990	980	970	990	980	970	990	980	970
% Additive in grease	1%	2%	3%	1%	2%	3%	1%	2%	3%

3.3.3 Moisture content of lithium stearate

The sample tube was heated and flushed with nitrogen gas through all line and tube to remove moisture. The lithium stearate was weighed, and inserted into the sample tube by a vacuum system. The sample tube was heated to reach 100 °C, and then nitrogen gas was flowed through the tube at 200 ml/min for 30 minutes.

Nitrogen gas with moisture was bubbled into a titrator tube containing Hydranol-solvent CM. The moisture of lithium stearate was determined by titration the moisture containing in Hydranol-solvent CM with Hydranol composite-5.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

3.3.4 Test method for cone penetration (JIS K 2220– 5.3 and ASTM D-217)

The sample grease was kept at room temperature. The sample was placed into a worker cup until it overflows (air is avoided by packing with a spatula). The worker cup was connected to a worker machine, then run to 60 strokes within 1 minute. The sample was prepared in the cup for testing. Any excess grease extending over the rim of the cup was scraped off by moving the spatula across the rim of the cup. The cup was placed on the penetrometer table (Figure 3.2). The indicator shaft was set at zero position, and then the tip of the cone was set to allow the surface of the grease at a center of the container. The release lever was pressed to allow the cone to penetrate the sample for 5 seconds, and then the indicator shaft was depressed until it was stopped. The penetration from the indicator scale was recorded.

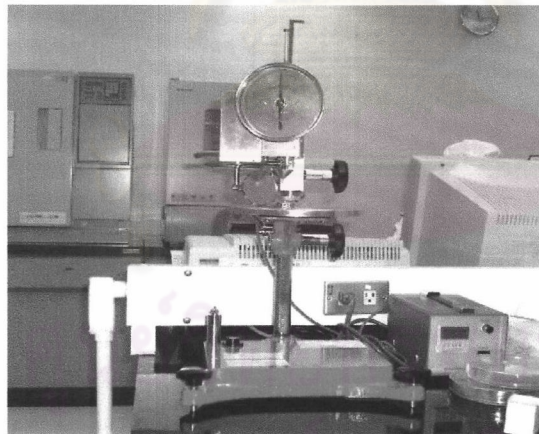


Figure 3.2 Penetrometer for measurement of grease penetration.

3.3.5 Test method for worked stability (ASTM D-217)

The grease, approximately 100 g, was placed in the worker. The apparatus was assembled by using the cover with 1/4 inch holes in the worker plate. The sample was worked for 60 strokes, and then penetration of grease was measured. (c.f. to 3.3.4).

The apparatus was reassembled and operated for 10,000 strokes by an electric motor (Figure 3.3). The sample grease was kept at room temperature to bring its temperature within the range of 20-30 °C, and then penetration of grease was measured again. The change in penetration between 60 and 10,000 strokes indicates the degree of mechanical stability of the grease.

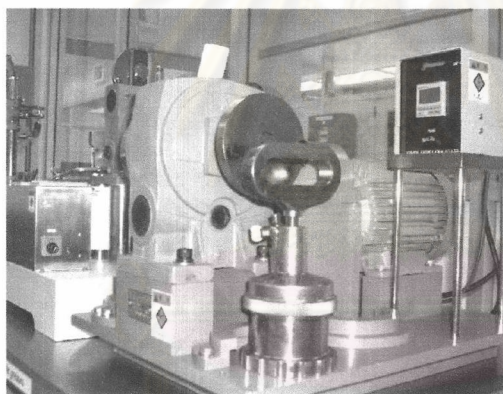


Figure 3.3 Grease worker for worked stability test.

3.3.6 Test method for dropping point (JIS K 2220–5.4 and ASTM D-566)

Grease samples were filled in a grease cup, and gently pressed the grease into the cup until a small amount is extruded at the small opening. The metal rod was used to cut the edge of grease from the cup, and a smooth film of grease should remain in the cup. The apparatus was assembled as shown Figure 3.4. The test thermometer was set by the tip of the thermometer bulb above the bottom of the grease cup when the apparatus is assembled. Another thermometer was placed in a silicone oil bath as same level as the test thermometer.

The silicone oil in the bath was stirred, and heated at a rate of 1-2 °C/min until the bath reached a temperature approximately 2 °C below the expected dropping point. The rate of heating was decreased as the temperature rise. The grease began flowing slowly through the orifice of the cup.

The temperature at which a drop of grease falls to the bottom of the cup is defined as the dropping point. The average of the reading temperature on two thermometers (thermometers in the sample cup and the oil bath) was considered as the dropping point of grease.

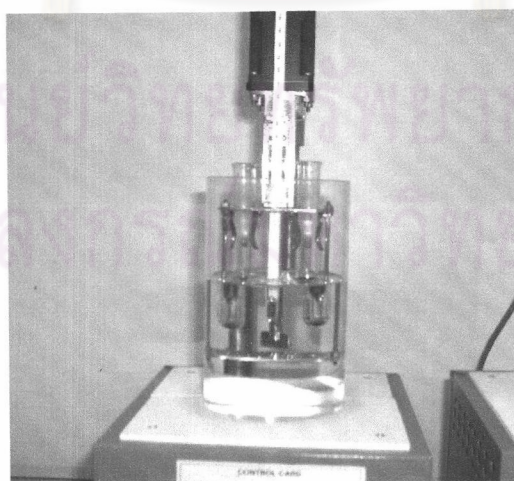


Figure 3.4 Apparatus for dropping point test of grease.

3.3.7 Test method for evaporation loss (JIS K 2220–5.6 and ASTM D-2595)

An empty sample cup was weighed at the room temperature. Then a test grease was filled in the sample cup with a spatula (avoid occlusion of air). The test grease was smooth, and the surface levels were set as same as the rim of the cup. The sample cup was weighed, and the net weight of the sample was recorded.

The sample cup was connected to a thermocouple and a heating device as shown in Figure 3.5. The cover was fasten, and then the temperature was adjusted to 99 °C. The air flow was set at the rate 2.58 ± 0.02 liter/minute.

After 22 hours, the assembled sample cup was removed from the heating device, and cooled down to room temperature. The sample cup was weighed, and the net weight of the sample was recorded. The loss in weight of grease was measured as evaporation loss of grease.

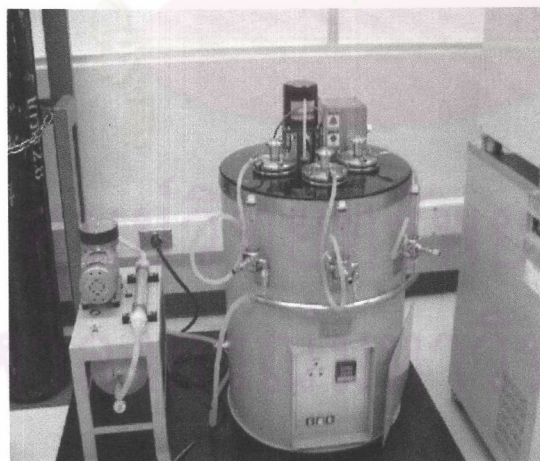


Figure 3.5 Apparatus for evaporation loss test of grease.

3.3.8 Test method for oil separation (JIS K 2220–5.7 and ASTM D-1742)

A 100-ml beaker was weighed, and a 60-mesh screen cone was assembled in a beaker with lid. The grease sample was packed into the 60-mesh screen cone supported in a tarred 100-ml beaker with lid (Figure 3.6). The beaker was placed in an oven at a constant temperature 100 °C for 24 hours. The beaker was removed from the oven and cooled in a desiccator. The 60-mesh screen cone was removed from the beaker, and the beaker was weighed.

An increase in weight of the beaker indicates the amount of oil bleeding or separation that occurred.



Figure 3.6 Apparatus for oil separation test of grease.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

3.3.9 Test method for oxidation stability (JIS K 2220- 5.8 and ASTM D-942)

The sample grease (4 g) was filled into a dish. The test grease was smooth, and the surface levels were set as same as the rim of the dish. Five dishes were placed in the dish holder, and placed in the Norma-Hoffman oxidation bomb (Figure 3.7). Oxygen pressure (0.69 MPa) was introduced into the bomb for 2 hours to check the leaking. If no leaks, the bomb was placed in an oil bath previously heated to 99 °C. As the pressure rised, oxygen pressure from the bomb was released until a constant pressure of 0.76 MPa. The oxygen pressure was observed, and recorded after 100 hours.

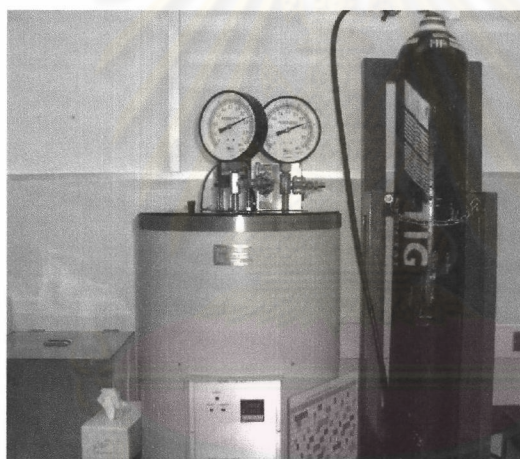


Figure 3.7 Apparatus for oxidation stability test of grease.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

3.3.10 Test method for corrosion preventive properties (ASTM D-1743)

A cleaned bearing was loaded with 20 mg of a grease sample. Bearing was run under a light thrust load for 1 minute to distribute the loaded grease in the bearings. The bearing was placed in a glass jar containing water 5 ml. The glass jar was capped with a screw top glass jar, and stored at 52 °C (Figure 3.8). After 48 hours, the bearing was cleaned, and examined for evidence of corrosion. Criterion for failure was the presence of any spot (1.0 mm or larger) in any direction.

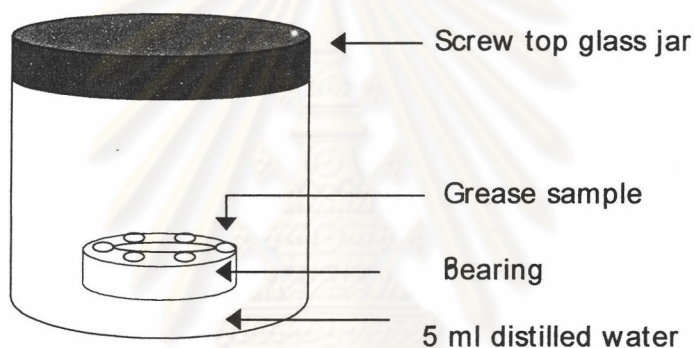


Figure 3.8 Apparatus for corrosion preventive property test of grease.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

The copper strip (12.5 mm wide, 50 mm long) was polished with 240-grit silicon carbide paper to remove all marks. The copper strip was immersed in hexane, and polished with 150-mesh silicon carbide paper then cleaned with cotton. The copper strip was immersed in a test tube containing sample grease. The test tube was placed in a constant-temperature oven at 100 °C for 24 hours. The strip was compared with ASTM Corrosion Standard.

3.3.12 Volatile organic compound (VOC) test

The grease (5 mg) was put in aluminum foil by controlled surface area about 2 mm x 5 mm. The foil was placed in a teflon chamber of Dynamic Headspace System (DHS), and baked at 85 °C for 3 hours. Helium gas was flowed to a chamber through an absorption tube (Carbotrap C and Carbotrap B packing material). After 3 hours, the absorption tube was inserted into the automatic thermal desorption (ATD 400), and then the volatile compounds in grease were analyzed by GC-MS [29-30] using the following conditions.

GC Conditions : The HP-5 Column (0.25 mm i.d. x 30 m, 0.25 µm film thickness) was used. Oven was set at 40 °C for 2 min with temperature ramp rate of 8 °C/min, until 240 °C for 13 min.

ATD Conditions : Oven of ATD was set at 380 °C and helium gas was flowed at rate 50 ml/min for 15 min.

DHS Conditions : Oven of DHS was set at 85 °C and helium gas was flowed at rate 50 ml/min for 3 hr.

3.3.13 Study of lithium soap structure by scanning electron microscopy

A small amount of grease was put on a slide glass (5 x 5 mm). The slide glass was dipped in hexane for 2 hours to remove the base oil. The thickener on slide glass was coated with a carbon rod by the carbon coating machine, and then analyzed by scanning electron microscopy (SEM) at magnification 20,000 to 50,000.



ศูนย์วิจัยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย