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APPENDICES

APPENDIX 1 Specification of grease for rolling bearing grade 1

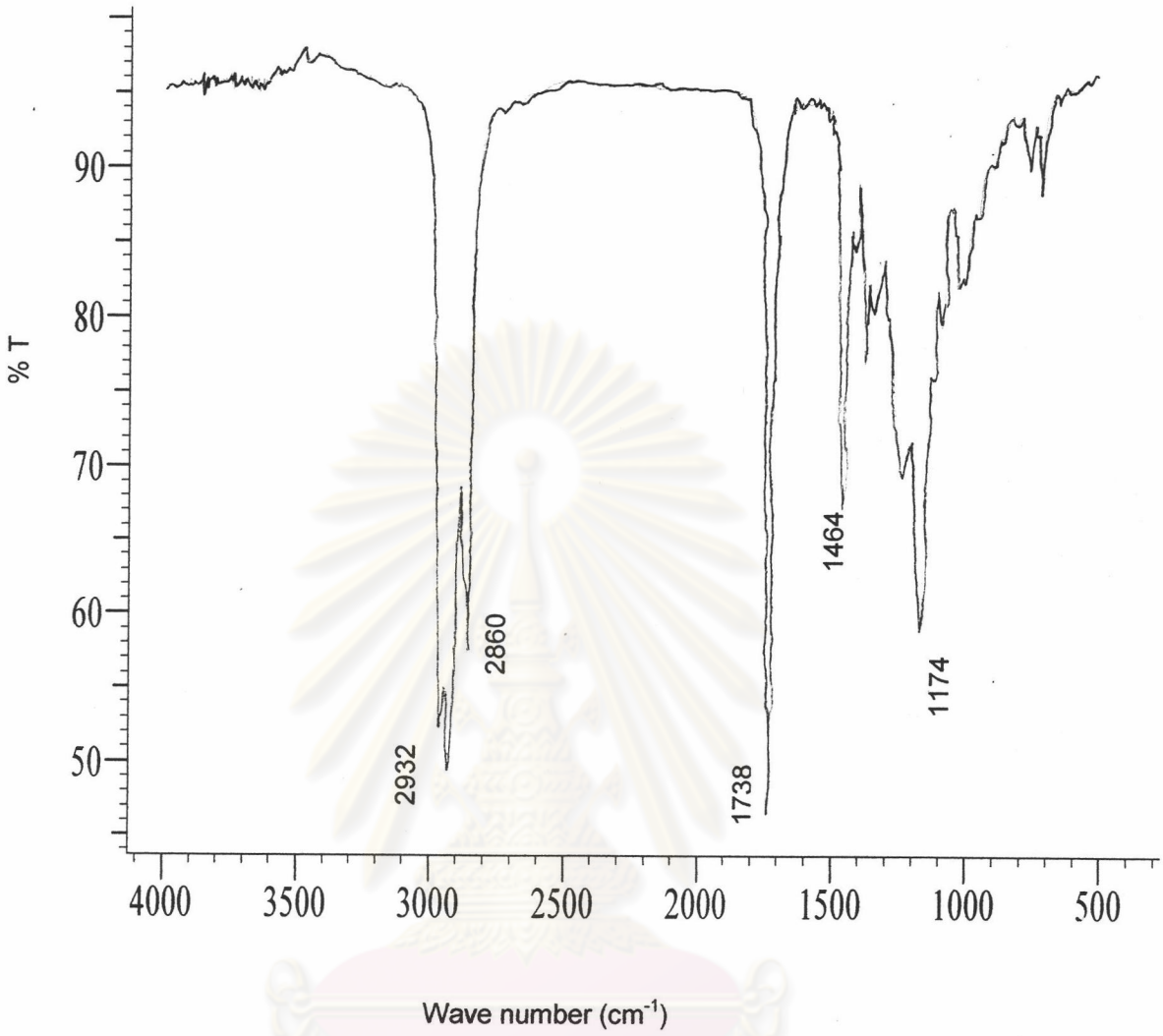
Grease for bearing grade 1, classified into 3 numbers according to a worked penetration range.

Item	Test method JIS K 2220	No. 1	No. 2	No. 3
Penetration at 60 strokes	5.3	310 to 340	265 to 295	220 to 250
Dropping point (°C)	5.4	170 min.	175 min.	175 min.
Copper corrosion (100 °C, 24 h)	5.5	No change to green or black color on copper plate		
Evaporation loss (99 °C, 22 h) mass%	5.6	2.0 max.	2.0 max.	2.0 max.
Oil separation (100 °C, 24 h) mass%	5.7	10 max.	5 max.	5 max.
Oxidation stability (99 °C, 100 h) MPa	5.8	0.069 max.	0.069 max.	0.069 max.
Worked stability penetration at 10,000 strokes	5.11	400 max.	375 max.	350 max.

Grease for rolling bearing shall be classified into 3 grades according to the use;

- grade 1 for general purpose (-20 °C to 100 °C)
- grade 2 for low temperature (-40 °C to 80 °C)
- grade 3 for a wide temperature range (-30 °C to 130°C)

APPENDIX 2 FTIR spectrum of lithium stearate



APPENDIX 3 Melting point and moisture content of lithium stearate

Lithium stearate	Test no.	Test items	
		Melting point (°C)	Moisture (%)
Commercial lithium stearate	1	206	0.16
	2	207	0.19
	3	207	0.20
	Average	207	0.18
Prepared lithium stearate	1	206	0.24
	2	206	0.26
	3	206	0.22
	Average	206	0.24

APPENDIX 4 Penetration of lithium grease at 0 and 60 strokes

Ratio of base oil and Li stearate (wt%)	Test No.	TMP-ester grease		Paraffin grease	
		0 stroke	60 strokes	0 stroke	60 strokes
80 : 20	1	201	206	166	170
	2	199	205	165	171
	3	201	205	167	170
	Average	200	205	166	170
82 : 18	1	221	224	190	194
	2	219	225	188	195
	3	221	225	188	193
	Average	220	225	189	194
85 : 15	1	249	254	221	223
	2	246	251	218	224
	3	246	252	217	225
	Average	247	252	219	224
90 : 10	1	298	303	283	289
	2	299	307	284	286
	3	302	304	283	286
	Average	300	305	283	287

APPENDIX 5 Dropping point of grease with 2 % additive

Base grease	Test no.	Dropping point (°C)			
		No additive	ZDDP	MDTC	DPA
TMP-ester grease	1	197	197	197	196
	2	197	196	197	196
	3	197	197	197	196
	Average	197	197	197	196
Paraffin grease	1	195	195	196	196
	2	195	195	197	196
	3	196	195	196	196
	Average	195	195	196	196
Commercial grease	1	196			
	2	195			
	3	195			
	Average	195			

APPENDIX 6 Evaporation loss of grease with 2 % additive

Calculate the evaporation loss of grease as follows:

$$\% \text{ evaporation loss} = \frac{W_S - W}{W_S} \times 100$$

Where W_S is the weight of a sample before test (g) and W is the weight of a sample after test (g). Evaporation loss of grease showed in table below.

Table A 6-1 Evaporation loss of prepared grease

Sample	Test no.	Weight of grease (g)		% Evaporation loss	Average
		Before test	After test		
TMP-ester grease with 2 % additive					
No additive	1	9.5123	9.3953	1.23	1.25
	2	9.4784	9.3590	1.26	
	3	9.7854	9.6631	1.25	
ZDDP	1	9.5101	9.3988	1.17	1.19
	2	9.4698	9.3562	1.20	
	3	9.4552	9.3270	1.19	
MDTC	1	9.6250	9.5066	1.23	1.22
	2	9.5500	9.4335	1.22	
	3	9.7411	9.6223	1.22	
DPA	1	9.5990	9.4800	1.24	1.23
	2	9.5013	9.3844	1.23	
	3	9.5733	9.4565	1.22	
Paraffin grease with 2 % additive					
No additive	1	9.5787	9.4743	1.09	1.10
	2	9.6302	9.5262	1.08	
	3	9.5470	9.4401	1.12	
ZDDP	1	9.7003	9.5917	1.12	1.11
	2	9.6564	9.5492	1.11	
	3	9.5840	9.4776	1.11	
MDTC	1	9.4789	9.3756	1.09	1.09
	2	9.5120	9.4093	1.08	
	3	9.5986	9.4930	1.10	
DPA	1	9.6587	9.5515	1.11	1.10
	2	9.5560	9.4509	1.10	
	3	9.7008	9.596	1.08	

Table A 6-2 Evaporation loss of commercial grease

Sample	Test no.	Weight of grease (g)		% Evaporation loss	Average
		Before test	After test		
Commercial grease	1	9.5877	9.4726	1.20	1.20
	2	9.6006	9.4864	1.19	
	3	9.592	9.4769	1.20	

APPENDIX 7 Oil separation value of grease with 2 % additive

Calculate the oil separation of grease as follows:

$$\% \text{ oil separation} = \frac{A}{B} \times 100$$

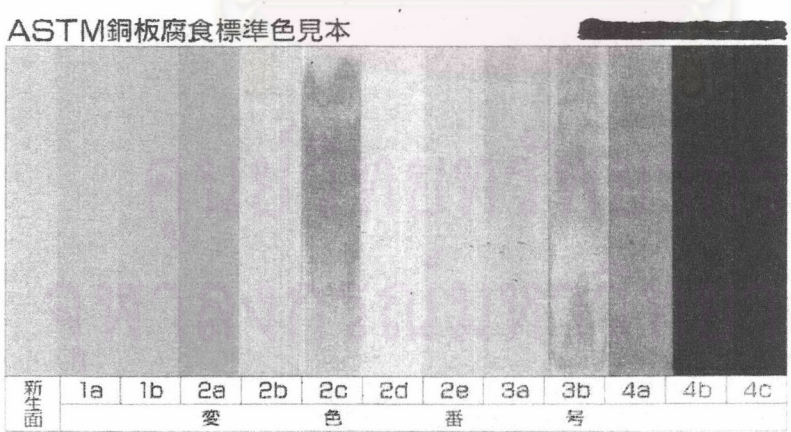
Where A is the weight of separated oil after test (g) and B is the weight of grease (g). Oil separation of grease showed in table below.

Table A 7-1 Oil separation (%) of prepared grease

Sample	Test no.	Weight of sample (g)		% Oil separation	Average
		Grease	Oil		
TMP-ester grease with 2 % additive					
No additive	1	10.1355	0.3335	3.29	3.32
	2	10.2472	0.3382	3.30	
	3	10.1479	0.3410	3.36	
ZDDP	1	10.3052	0.3442	3.34	3.31
	2	10.1988	0.3376	3.31	
	3	10.2094	0.3359	3.29	
MDTC	1	10.1749	0.3388	3.33	3.32
	2	10.2987	0.3378	3.28	
	3	10.3650	0.3472	3.35	
DPA	1	10.2110	0.3349	3.28	3.29
	2	10.1961	0.3344	3.28	
	3	10.2330	0.3377	3.30	
Paraffin grease with 2 % additive					
No additive	1	10.1145	0.3307	3.27	3.29
	2	10.2350	0.3357	3.28	
	3	10.1978	0.3375	3.31	
ZDDP	1	10.2478	0.3341	3.26	3.28
	2	10.3232	0.3386	3.28	
	3	10.0988	0.3323	3.29	
MDTC	1	10.2550	0.3323	3.24	3.25
	2	10.2871	0.3354	3.26	
	3	10.2468	0.3330	3.25	
DPA	1	10.2700	0.3379	3.29	3.26
	2	10.1790	0.3298	3.24	
	3	10.2685	0.3348	3.26	
Commercial grease					
	1	10.0945	0.3311	3.28	3.29
	2	10.1469	0.3338	3.29	
	3	10.278	0.3392	3.30	

APPENDIX 8 ASTM standard for copper corrosion test

Classification	Designation	Description
1a	Slight tarnish	Light orange, nearly to freshly polished strip
1b	Slight tarnish	Dark orange
2a	Moderate tarnish	Claret red
2b	Moderate tarnish	Lavender
2c	Moderate tarnish	Multicolor with lavender blue or silver, overlaid on claret red
2d	Moderate tarnish	Silvery
2e	Moderate tarnish	Brass or gold
3a	Dark tarnish	Magenta overcast on brassy strip
3b	Dark tarnish	Multicolor with red and green
4a	Corrosion	Transparent black, dark gray or brown
4b	Corrosion	Graphite or lusterless black
4c	Corrosion	Glossy or jet black



APPENDIX 9 Volatile organic compounds of lithium grease

Calculation of volatile organic compounds from grease as follows

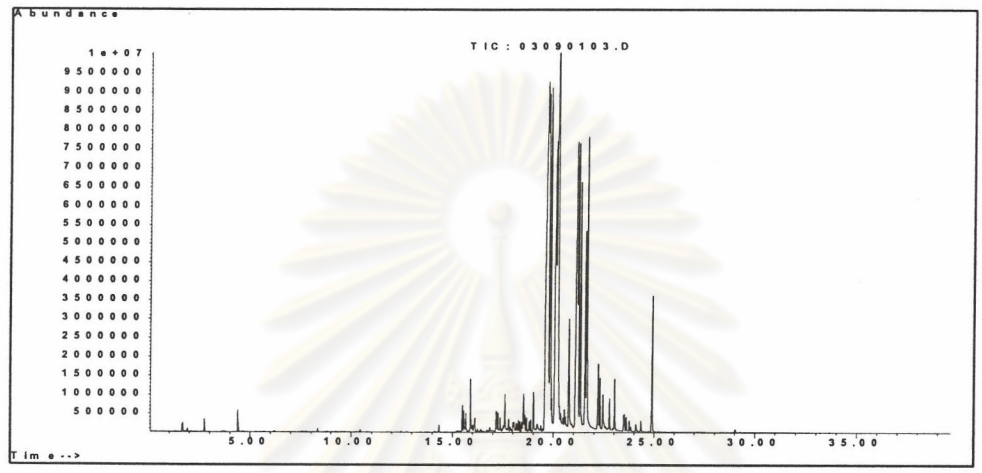
$$\text{Outgas (ng/mg)} = \frac{\text{total peak area of grease x concentration of hexadecane}}{\text{peak area of hexadecane x weight (mg) of grease}}$$

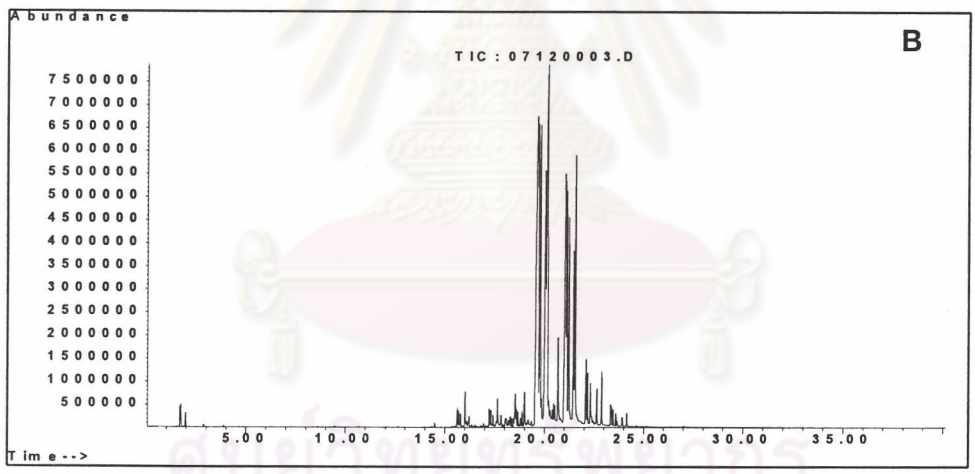
Area of standard hexadecane at 1,000 ng was found to be equal to 30,573,014 for this experiment. The amount of volatile organic compounds (ng/mg) from lithium grease showed in table below

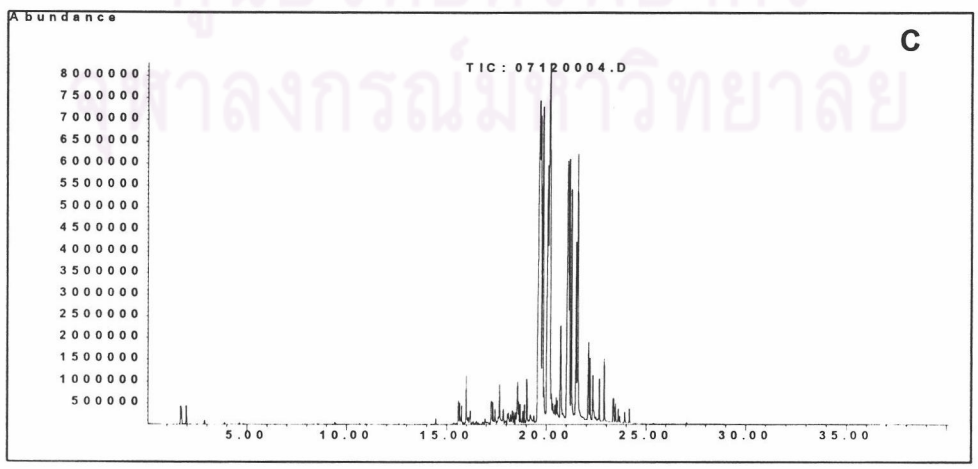
Sample	Test No.	Grease (mg)	Total area	Outgas (ng/mg)	Average (ng/mg)
Lithium-Paraffin oil grease with 2% additive					
ZDDP	I	5.23	714,836,210	4,471	4,513
	II	5.36	746,598,314	4,556	
MDTC	I	5.12	682,872,663	4,362	4,280
	II	5.5	705,698,431	4,197	
DPA	I	5.48	739,984,541	4,417	4,341
	II	5.69	741,895,647	4,265	
Lithium-TMP oil grease with 2% additive					
ZDDP	I	5.11	1,086,524,131	6,955	6,813
	II	5.67	1,156,389,782	6,671	
MDTC	I	5.57	1,134,787,451	6,664	6,737
	II	5.33	1,109,784,325	6,810	
DPA	I	5.42	1,145,897,544	6,915	6,854
	II	5.29	1,098,754,623	6,794	
Commercial lithium-ester grease					
	I	5.38	977,648,712	5,944	5,833
	II	5.66	990,356,984	5,723	

APPENDIX 10 GC-MS chromatograms of TMP ester grease

GC-MS chromatograms of volatile organic compounds from TMP ester grease with 2 % additives of DPA (A), ZDDP (B) and MDTC (C).

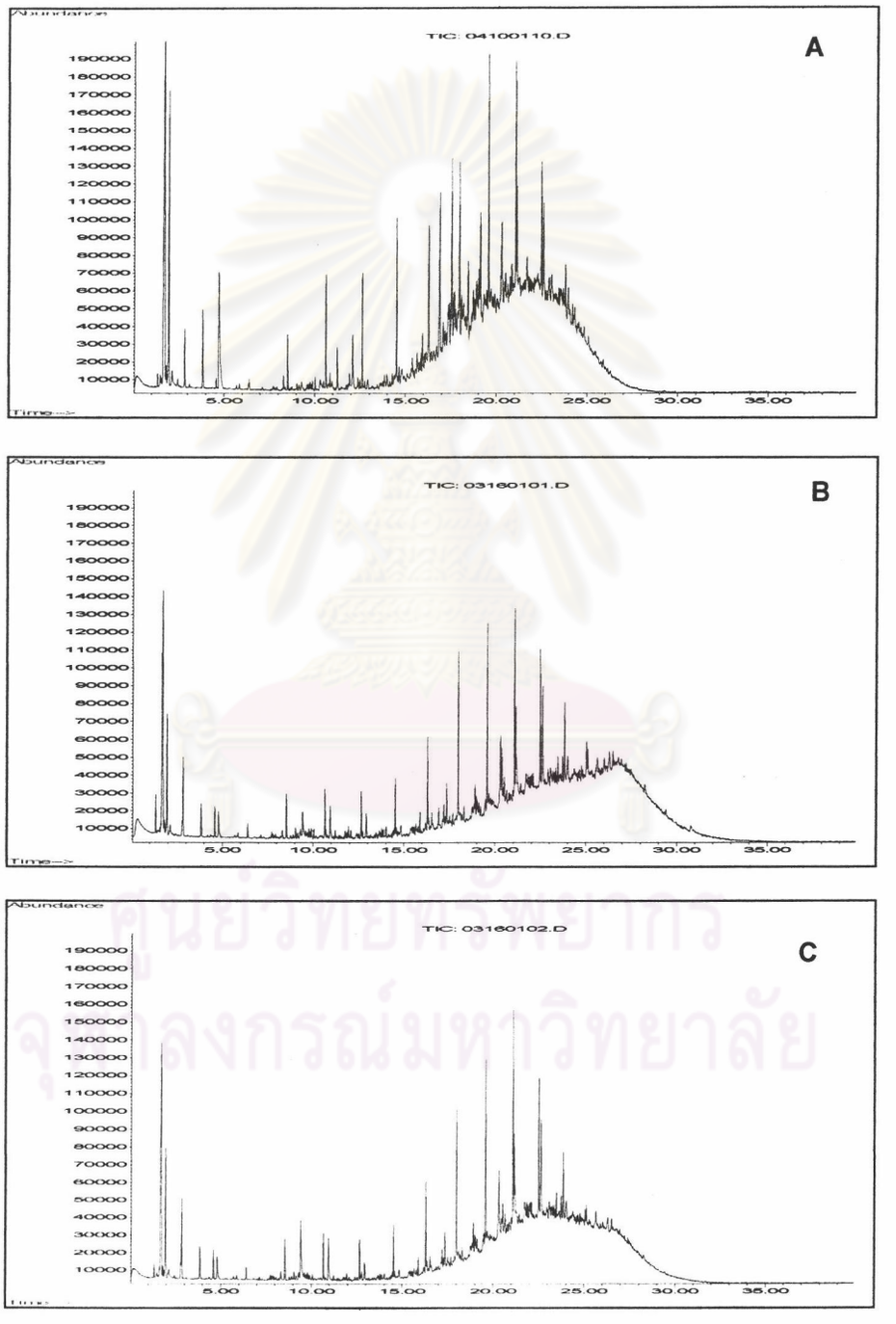




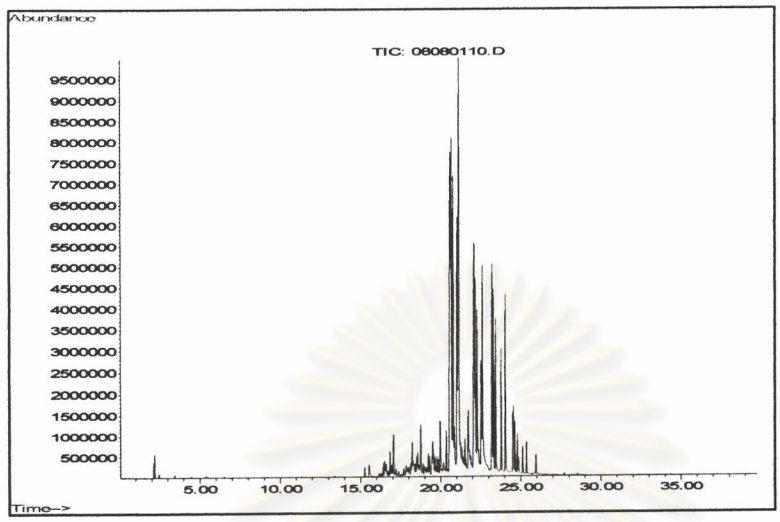


APPENDIX 11 GC-MS chromatograms of paraffin grease

GC-MS chromatograms of volatile organic compounds from paraffin grease with 2% additives of DPA (A), ZDDP (B) and MDTC (C).



APPENDIX 12 GC-MS chromatogram of commercial lithium grease



APPENDIX 13 NLGI number & penetration of grease at 60 strokes

NLGI consistency numbers and penetration number of grease worked at 60 strokes and measured by ASTM D-217.

NLGI Number	Penetration at 25 °C, 60 strokes
000	445-475
0	400-430
0	355-385
1	310-340
2	265-295
3	220-250
4	175-205
5	130-160
6	85-115

APPENDIX 14 Properties of base oils and additives

Table A 14-1 Properties of base oils

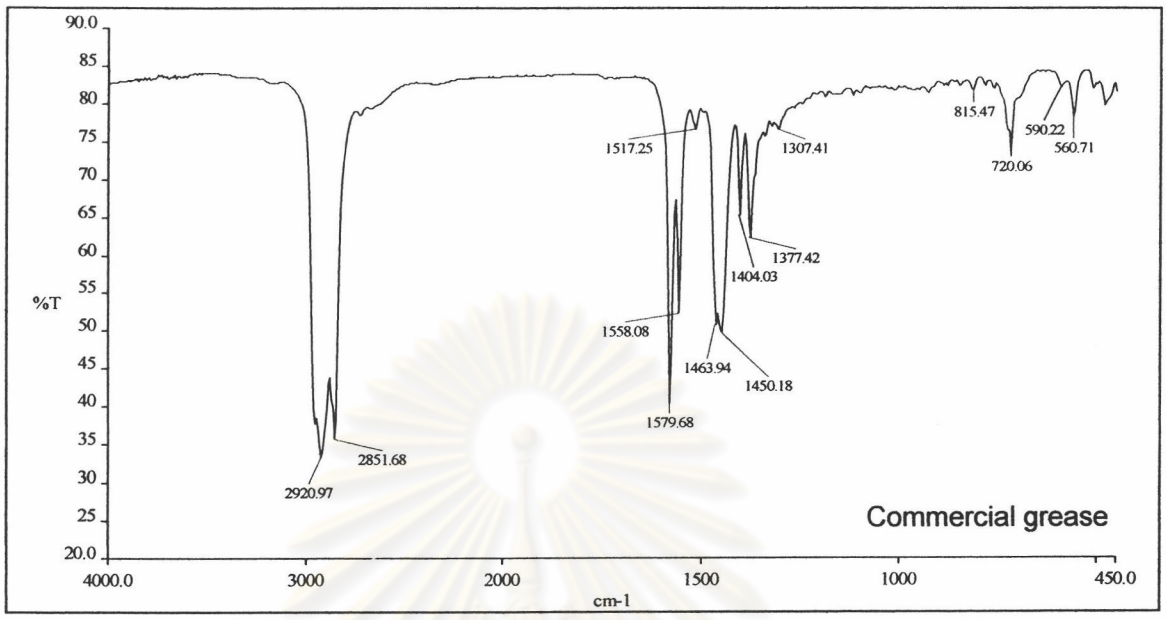
Properties*	Base oil	
	TMP ester oil	Paraffin oil
Viscosity		
40 °C (cSt)	17.6	40.1
100 °C (cSt)	3.8	7.3
Pour Point (°C)	-40	-29
Flash Point (°C)	230	226
Density @ 20 °C (g/cm^3)	0.868	0.859

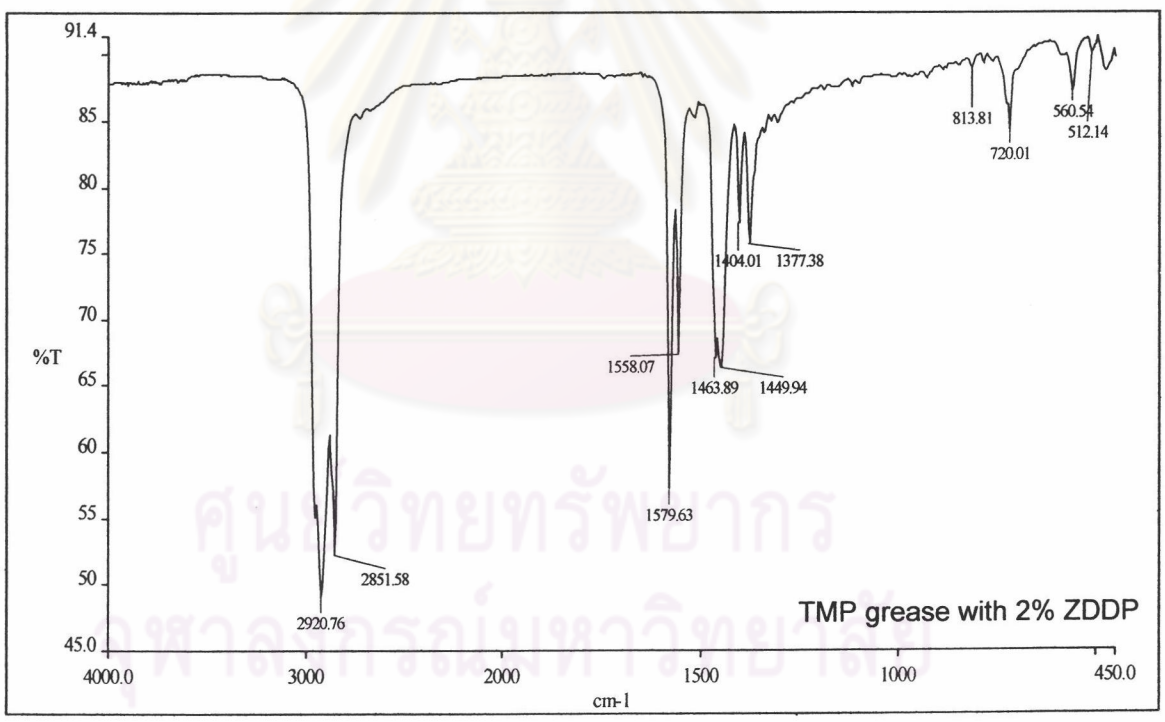
* Test at Laboratory of Minebea Thai Ltd.

Table A 14-2 Properties of additives

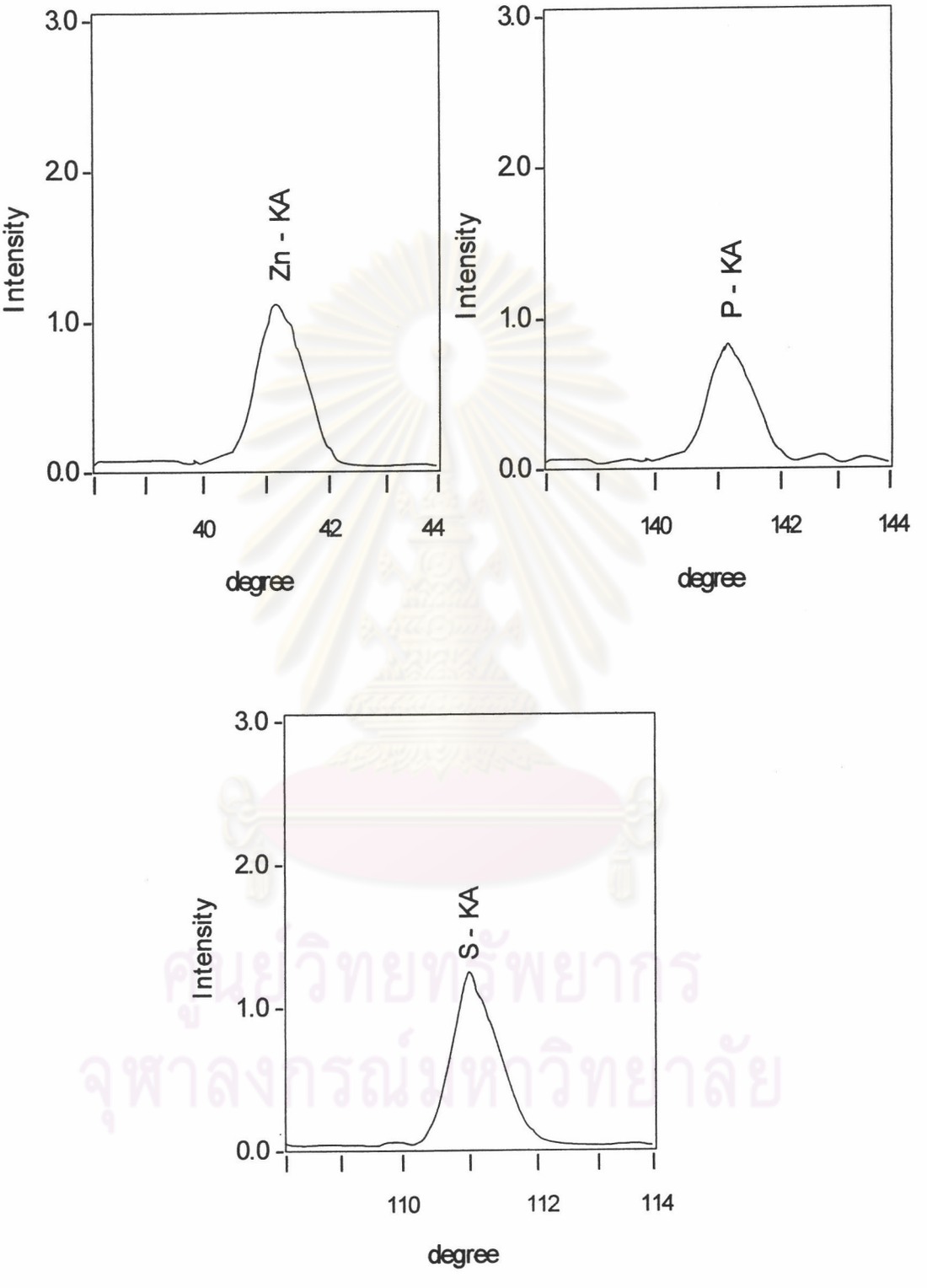
Properties	Additives		
	ZDDP	MDTC	DPA
Color	Reddish Brown	Dark greenish brown	Light yellow
Density (g/cm^3) @20°C	1.01	1.02	0.98
Flash point (°C)	182	183	185

APPENDIX 15 FT-IR spectrum of lithium grease with additive

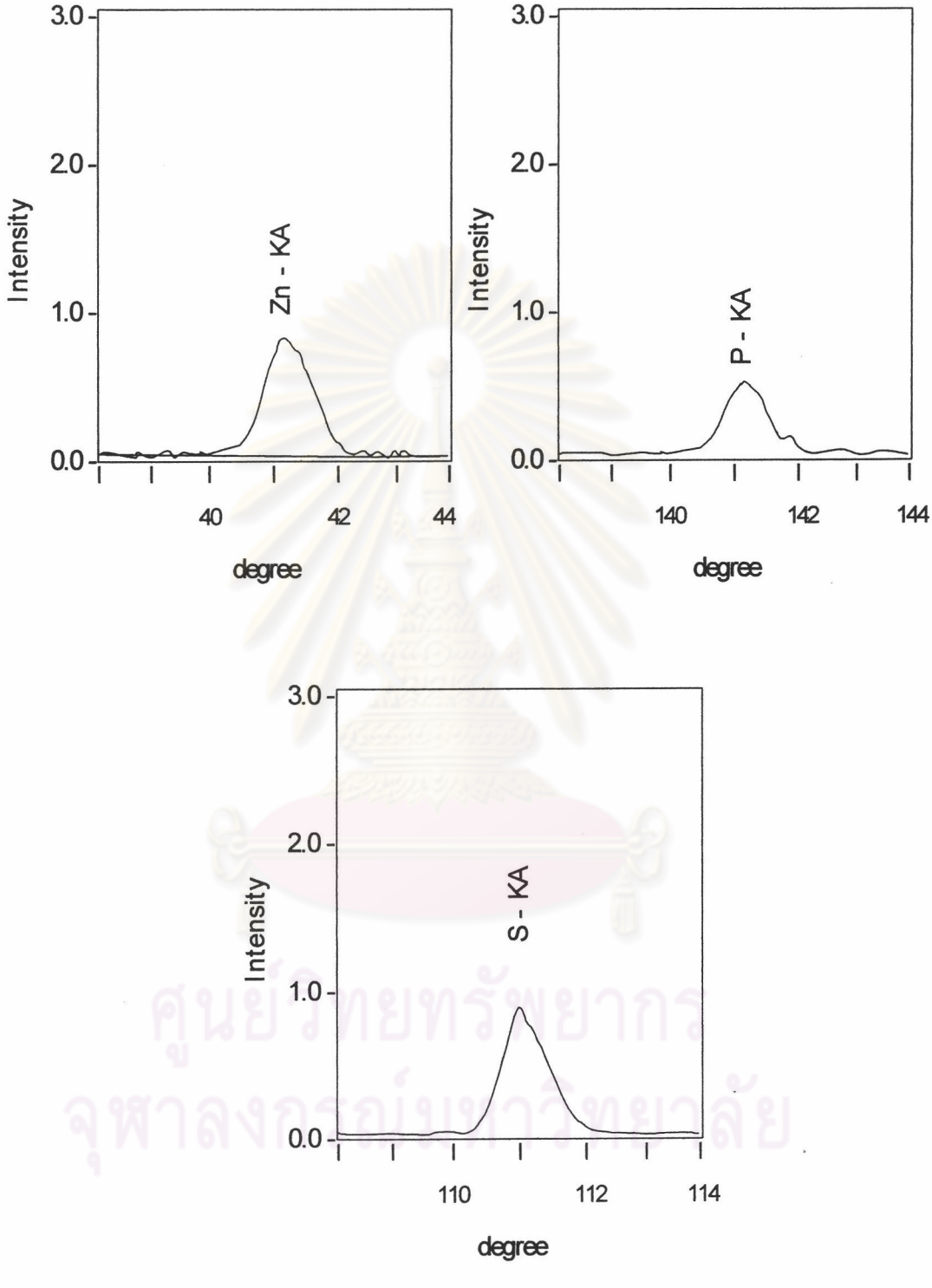




APPENDIX 16 XRF spectrum of commercial grease



APPENDIX 17 XRF spectrum of TMP grease with 2% ZDDP



VITA

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