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SINTERING-RESISTANCE AND ABRASION-RESISTANCE CAPACITY OF PHOSPH--ETC(U)

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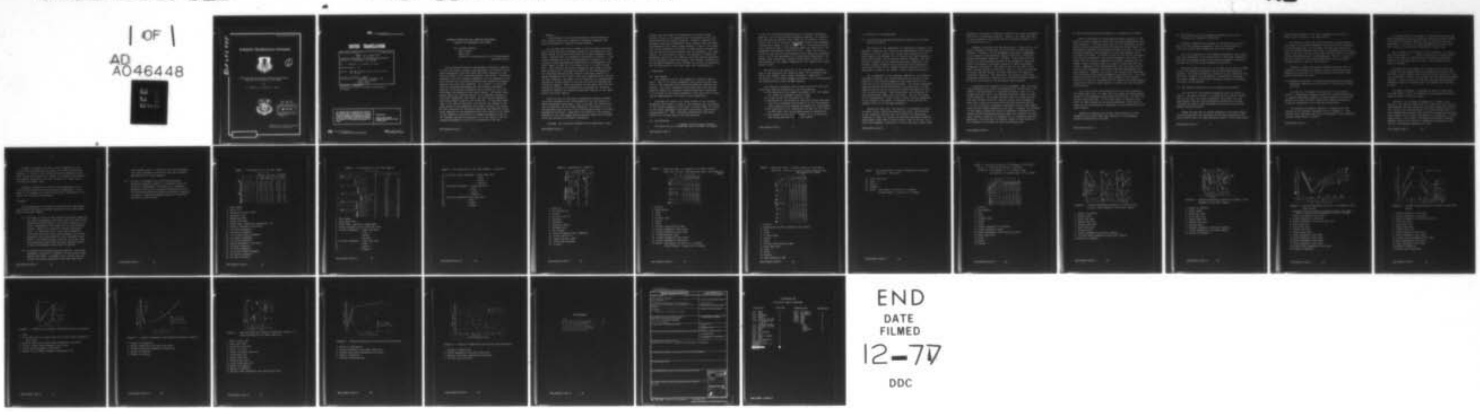

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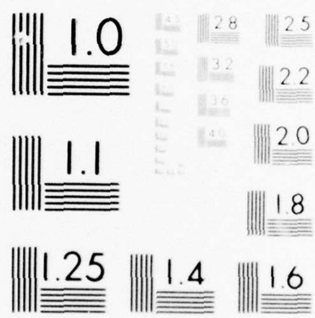

MICROCOPY RESOLUTION TEST CHART
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SINTERING-RESISTANCE AND ABRASION-RESISTANCE CAPACITY OF PHOSPHORIC ACID ESTER

by

Y. Kurachi, S. Hirano, K. Yano

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By: Y. Kurachi, S. Hirano, K. Yano

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FOREIGN TECHNOLOGY DIVISION
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SINTERING-RESISTANCE AND ABRASION-RESISTANCE CAPACITY OF PHOSPHORIC ACID ESTER

BY: Yoshiaki Kurachi
Shuji Hirano
Kakio Yano
Division of Lubricating Oil of The Merchandise Research Center

The sintering-resistance and abrasion resistance capacity of both the neutral, acidic alkyl-phosphoric acid ester and the acidic-phosphoric acid ester amin was fully evaluated or measured by means of the fourfold globular test device. Scientific research on how the chemical composition of the phosphoric acid ester affects the sintering resistance and abrasion resistance capacity has also been conducted. As a result, it was found that the sintering resistance and the abrasion resistance capacity of the acidic phosphoric ester turned out to be the best among the phosphoric acid ester family. The acidic phosphoric ester amin salt was proven to be slightly inferior to the acidic phosphoric acid ester, and the neutral phosphoric ester was almost as good as the first two. It was also found that in the case of amin salt, the shorter the length of the alkyl group of the phosphoric acid ester and amin, the better. We also evaluated the sintering resistance and abrasion resistance capacity of the phosphoric acid ester family on the basis of the formation inclination of inorganic-phosphoric acid in the chemical process of pyrolysis, or on the basis of how the inorganic-phosphoric acid is to be absorbed by the iron.

1 Preface

The phosphoric acid ester having been used extensively since ancient times as an abrasion resistance compound, has been investigated or studied by many scholars.

It has been said that in terms of the sintering resistance capacity and abrasion resistance capacity, the phosphoric acid ester claims to be much better than the neutral phosphoric acid ester. Granted that the phosphoric acid ester is better, it has a few problems such as the acid-instability, erosion, etc. On account of these problems mentioned above, the phosphoric acid ester cannot be used as it is unless it is to be used as amin salt. Thus recently the acidic phosphoric acid ester is being used as amin salt. Although the result of the experiments on the sintering resistance and abrasion resistance capacity of acidic phosphoric acid ester - amin salt conducted by Dr. Forbes has been reported, the report is not conclusive or comprehensive because Dr. Forbes seemed to use only one kind of annexing-concentration sample. Thus the authors of this paper took the following two points into consideration concerning the sintering resistance capacity or abrasion resistance capacity of the phosphoric acid ester family.

First of all, we have made a comparative analysis between the acidic phosphoric acid ester-amin salt and the neutral or acidic phosphoric acid ester. That is to say, the characteristics of amin salt of the acidic phosphoric acid ester is to be distinguished from the one of the conventional neutral or acidic phosphoric acid ester. Especially the acidity or the chemical reaction of the test sample was extensively taken into consideration for testing its abrasion resistance.

Secondly, the so-called phosphoric iron hypothesis, which insists that the reason the phosphoric acid ester causes abrasion is due to the formation of the phosphoric iron, has conventionally been widely accepted. However, currently the phosphoric acid-iron hypothesis, which insists that the reason the phosphoric acid ester causes abrasion is due to the formation of the phosphoric acid-iron, is being widely accepted by the scholars and is more popular than the phosphoric iron hypothesis. It was found out that in the process of forming the phosphoric acid-iron, the chemical reactions such as pyrolysis of the phosphoric acid ester family, the chemical formation of the inorganic phosphoric acid through the process of hydrolysis, and the absorption of iron were taken into consideration.

2 Experiment

2-1 Test Sample

The properties of the test sample are listed in Table 1 and 2. All of the acidic phosphoric acid ester and amin salt used in this experiment are laboratory-prepared chemical substances. The acidic phosphoric acid ester amin salt was prepared by adding the carefully prepared alkyl-amin equivalent to the amount of the acidic phosphoric acid ester and by churning for approximately 4-8 hours at room temperature.

Although the value of the total acidity of the neutral phosphoric acid ester is close to zero meaning that the purity of the neutral phosphoric acid ester is almost 100%, the purity of the acidic phosphoric acid ester containing a monoester is approximately 80 - 90%. Carefully manufactured high-grade neutral oil 150 was used as the base gasoline.

2-2 Lab-Technique

The Shell Ball E P Tester (1500rpm, friction speed 56m/sec) was used to measure the degree of both the sintering-resistance and the abrasion-resistance capacity. As to the abrasion-resistance capacity, the measurement was made by adding a load (30kg) for 30 minutes, and as a result, the trace of abrasion was measured within the phosphate concentration range of 0.01 ~ 0.20 wt.% As to the sintering-resistance capacity, when it was measured under the condition of the phosphate concentration range from 0.10 - 0.20%, the initial sintering load and the load absorption were taken into consideration. As to the measurement of the pyrolytic temperature, the Shimazu type differential thermal balance MTG-11 was used. It was measured in the oxygen atmosphere of the temperature rising speed 5°C/min.

The survey of the formation of the inorganic phosphoric acid by the pyrolysis was conducted under the following conditions: test sample: bulk, oxygen atmosphere (3ℓ/hr) 190° C. The amount of the formed inorganic phosphoric acid was measured by the neutralization titer and NMR analysis.

The chemical absorption test in which the JIS K 2514 test device was used was condcuted in the following way:

a) The deoxidized iron powder was added to the test sample oil (100g) of phosphate content 0.10%.
b) It was churned for 30 minutes at 170° C.
c) The amount of the phosphoric acid ester family was measured as the phosphate concentration by the flourescence x-ray analysis after washing thoroughly the iron powder.
d) The condition of the temperature was kept at 170° C which is 20° C lower than the minimum pyrolytic temperature.
e) The remains of the resolved sample is to be prevented from being absorbed into the iron powder.

3 The Result of the Experiment

3-1 Concerning the Abrasion-Resistance Capacity of the Phosphoric Acid Ester

The result of the experiment is indicated in diagram 1 & 2. Diagram 1 indicates the result of the measurement on the amount abrasion of butyl, octyl, and 2-ethyl hexyl phosphoric acid ester family, and diagram 2 on the lauryl and oleyl phosphoric acid ester family. As is obvious from the diagrams, the comparative analysis on the abrasion-resistanve capacity of the neutral, acidic phosphoric acid ester and acidic phosphoric acid ester (butyl, octyl, lauryl, stearyl amin salt, etc) is explained in terms of the effect of each chemical structure.

As is indicated in the diagrams, in terms of the abrasion-resistance capacity, the acidic phosphoric acid ester is far superior to the neutral phosphoric acid ester which is not effective at all in preventing abrasion. The acidic phosphoric acid ester shows the minimum value of abrasion with a little additive amount and as the additive amount increases the abrasion is also increased. This inclination can noticeably be seen particularly in the butyl acid ester. And as the length of the alkyl-group of butyl, octyl, lauryl, and the phosphoric ester increases, the amount of abrasion, after gaining the minimum value, is to increase gradually.

Among each amin salt, the abrasion is increased in order of butyl, octyl, lauryl and stearyl amin salt, and therefore we can establish the rule that the shorter the alkyl-group (radical) of amin, the better the abrasion-resistance capacity. Particularly butyl amin is worth notice, that is to say, as is clear from diagram 1, amin salt of each octyl and 2-ethyl hexyl phosphoric acid ester is almost as effective as acidic phosphoric acid ester in resisting abrasion. However, the butyl amin salt of butyl phosphoric acid ester was not evaluated because it is not soluble by the oil.

Diagram 3 indicates the following facts. First of all, the neutral phosphoric acid ester has no effect on resisting the abrasion. Secondly, acidic phosphoric acid ester, regardless of the length of alkyl-radical (group) exhibits an excellent abrasion-resistance capacity. Thirdly, it was found that the shorter the alkyl-radical (group) of the phosphoric ester, the better the abrasion resistance capacity of amin salt(acidic phosphoric acid ester). The same is true in the case of the alkyl radical (group) of amin salt, that is to say, the shorter the alkyl radical (group), the more effective the abrasion-resistance capacity. The reason why the acidic 2-ethyl hexyl phosphoric acid ester exhibits such a peculiar demeanor seems to be due to the effect of the horizontal chain-radical.

According to Dr. Forbes and his colleagues, when the acidic butyl phosphoric acid ester is compared with its amin salt, the former is less effective in resisting abrasion, and if it is changed into amin salt, its abrasion-resistanve power is extensively improved. Dr. Forbes contends that the reason why the amin salt is more effective than the acidic butyl phosphoric acid ester is due to the fact that the amin salt was used as it is as the abrasion resistance substance without being separated from the acidic phosphoric acid ester. However, the authors of this paper contend that such an experiment conducted by Dr. Forbes and his colleagues was done under the condition of phosphate content 0.12% or 0.15 wt% which is an extremely high concentration, and therefore Dr. Forbes' report or test result is not relevant to the issue we have been dealing with.

3-2 The Sintering-Resistance Capacity of Phosphoric Acid Ester

Table 4 indicates the sintering-resistance capacity of the neutral, acidic phosphoric acid ester and acidic phosphoric acid ester amin salt. The top figure of each line indicates the phosphate content concentration 0.10 wt%, and the bottom figure in parenthesis indicates the phosphate content 0.20 wt%. The initial sintering load and load absorption of the base gasoline (oil) are 45kg, and 112kg. In terms of value of the initial sintering load and load absorption, the acidic phosphoric acid ester is higher than the neutral phosphoric acid ester, meaning that the former is more effective than the latter in resisting the sintering. In terms of the load-absorption, both the octyl and the 2-ethyl hexyl phosphoric acid ester exhibits approximately the same capacity. However, in terms of the initial load sintering, both the neutral and acidic phosphoric acid ester are a few units higher than the other (2-ethyl hexyl phosphoric acid ester). From this it must be concluded that in terms of the alkyl-radical (group) the vertical chain-radical is better than the horizontal chain-radical.

As to the initial load sintering of the acidic phosphoric acid ester amin salt, it seems that the longer the alkyl-radical (group) of the phosphoric acid ester and the amin, the lower the initial load sintering. On the other hand, it exhibits just about the same results as the acidic phosphoric acid ester in terms of the load absorption regardless of the difference of the alkyl-radical.

Diagram 4 indicates the initial load sintering of each phosphoric acid ester under the condition of the phosphate content concentration 0.10 wt%.

3-3 The Formation of the Inorganic Phosphoric Acid by the Pyrolysis of Phosphoric Acid Ester

Diagram 5 indicates the results of the survey on the formation of the inorganic phosphoric acid by the pyrolysis of butyl and 2-ethyl hexyl phosphoric acid ester family.

As to the formation of the inorganic phosphoric acid, under such test conditions it is limited only to the acidic phosphoric acid ester excluding entirely the neutral phosphoric acid ester and amin salt of the acidic phosphoric acid ester.

From the result of the test, it is safe to say that in terms of both the abrasion-resistance and the sintering-resistance capacity, the acidic phosphoric acid ester is more effective than the other. However, as the additive concentration increases, the acidic phosphoric acid ester inversely creates the abrasion on account of its erosion.

3-4 The Chemical Absorption of the Phosphoric Acid Ester

The results of the chemical absorption test is listed in Table 6. As shown in Table 6, in terms of the chemical absorption by the iron, the acidic phosphoric acid ester is a few times higher than the neutral phosphoric acid ester, and particularly the acidic butyl phosphoric acid ester exhibits tremendous absorption power.

Among the amin salt of acidic phosphoric acid ester family, all amin salts with the exception of the butyl phosphoric acid ester exhibit about the same absorption power of the acidic ester.

(The absorption power of the butyl phosphoric acid ester is smaller than the rest of the amin salts.)

The reason the amin salt has just about the same absorption power of the acidic phosphoric acid ester may be explained in the following way. On one hand, the amin salt coming off from the acidic phosphoric acid ester, is absorbed onto the surface of the iron. On the other hand, the amin cation is absorbed either electrostatically or overlappingly. Thus it is safe to say that there is a very little possibility of the covelant bond-absorption of amin by the electron.

Therefore, these test results indicate the fact that in terms of the structure of chemical action, neutral and acidic phosphoric acid ester is a little different from acidic phosphoric acid ester amin salt.

3-5 Comparative Analysis of Sintering-resistance (and Abrasion-resistance Capacity) and the Result of the Physical Properties Test

In order to investigate the structure of the chemical action, a comparative analysis between the sintering-resistance or abrasion-resistance capacity of these phosphoric acid ester families and their physical properties (chemical absorption, pyrolysis temperature, etc.) was done effectively.

The abrasion-resistance capacity is compared with the results of the chemical absorption test in diagram 6. (The vertical line indicates the traces of abrasion in the phosphate-content concentration 0.10wt%.)

As shown clearly in Diagram 6, although both neutral and acidic phosphoric acid ester exhibit a good correlation between the abrasion-resistance capacity and the amount of chemical absorption, in the case of acidic phosphoric acid ester amin salt, there is a very little correlation between them.

For the sake of the reader's future reference, the base of amin which should be used to neutralize the acidic phosphoric acid ester is compared with the abrasion resistance capacity of each amin salt of acidic octyl phosphoric acid ester in Diagram 7.

Although the test samples are limited, it was found that in terms of these samples such as di-phenyl amin, phenyl amin (anilin), phenyl di-methy amin, and cyclo-hexyl amin salt, there is a correlation between the amin base and the abrasion-resistance capacity. Thus it is safe to say that the abrasion-resistance capacity of amin salt depends upon the hydrogen bond strength of amin.

As shown in Diagram 7, although the bases of alkyl amin are fixed ($pKa \fallingdotseq 10.6$), the effects of the abrasion-resistance capacity exhibit different values.

Therefore, as is shown in Diagram 6, in terms of the amount of the chemical absorption, the alkyl amin salt exhibits approximately the same amount of the acidic phosphoric acid ester. Hence, the solid structure of the amin cation which is electrostatically absorbed onto the acidic phosphoric acid ester anion seems to have a great influence on the formation of lubricating tunic. As a result, the abrasion-resistance capacity depends rather on the length of alkyl-group (radical) of amin than on the chemical absorption of iron.

Diagram 8 indicates the side by side comparison of the initial load sintering and the amount of chemical absorption. Although, in terms of the neutral and acidic phosphoric acid ester, there are correlations between the loading sintering and the chemical absorption, in terms of amin salt, there are no correlations between them.

Diagram 9 indicates the side by side comparison of the initial load-sintering and the pyrolytic temperature. As is shown in the diagram, the lower the pyrolytic temperature, the higher the initial load-sintering.

4 Summary

The following are the results of the survey on the sintering-resistance/abrasion-resistance capacity of the alkyl phosphoric acid ester family:

(1) Although, in terms of the abrasion-resistance capacity, the neutral phosphoric acid ester does not exhibit the abrasion-resistance power, the acidic phosphoric acid ester, regardless of the length of the alkyl-radical (group), exhibits an excellent abrasion-resistance power. As mentioned above, the acidic phosphoric acid ester amin salt being ranked between the neutral phosphoric acid ester and the acidic phosphoric acid ester in its effectiveness, exhibits the better abrasion-resistance power when the alkyl-radical (group) of amin and phosphoric acid ester is shorter.

(2) In terms of sintering-resistance capacity, the acidic phosphoric acid ester is superior to the neutral phosphoric acid ester. In terms of the initial load sintering, the acidic phosphoric acid ester amin salt

ranks between them. In terms of the load absorption, the acidic phosphoric acid ester amin salt exhibits approximately the same capacity.

(3) We have investigared the sintering-resistance/abrasion-resistance capacity in connection with the pyrolytic temperature, the formation of inorganic phosphoric acid and the chemical absorption by iron. The sintering-resistance/abrasion-resistance capacity, in terms of absorption, depends greatly on the solid structure of amin cation.

TABLE 1 - The Properties of the Test Sample

表1 実験試料の性状

1 実験試料	2 純度 (%)	3 リン分 (wt%) 4 分析値	5 計算値	6 全酸価 (mgKOH/g)	7 一次熱分解温度 (°C)
8 中性リン酸エステル					
9 トリ-ブチルホスフェート	100	11.8	11.6	0.00	220
10 トリ-オクチル 〃	100	7.1	7.2	0.03	230
11 トリ-2-エチルヘキシル 〃	100	7.3	7.2	0.01	215
12 トリ-ラウリル 〃	100	5.0	5.1	0.02	230
13 トリ-オレイル 〃	100	3.8	3.7	0.01	255
14 酸性リン酸エステル					
15 ジ-ブチルホスフェート	80	15.8	14.7	308	190
16 ジ-オクチル 〃	87	9.7	9.6	178	200
17 ジ-2-エチルヘキシル 〃	88	9.9	9.6	176	190
18 ジ-ラウリル 〃	86	6.8	7.1	122	225
19 ジ-オレイル 〃	84	4.7	5.2	83	210

1 Test Sample
2 Purity (5)
3 Phosphate content (wt%)
4 Analysis value
5 Measuring value
6 Total acid value
7 First order pyrolytic temperature (°C)
8 Neutral phosphoric acid ester
9 tri-butyl phosphate
10 tri-octyl phosphate
11 tri-2-ethyl hexyl phosphate
12 tri-lauryl phosphate
13 tri-oleyl phosphate
14 Acidic phosphoric acid ester
15 di-butyl phosphate
16 di-octyl phosphate
17 di-2-ethyl hexyl phosphate
18 di-lauryl phosphate
19 di-oleyl phosphate

TABLE 2 - The Properties of the Test Samples

表2 実験試料の性状

1 実験試料	2 全酸価(mgKOH/g)	3 一次熱分解温度(°C)
4 酸性リン酸エステルアミン塩		
5 ジ-ブチルホスフェート―ブチルアミン塩	219	190
6 ―オクチル 〃	174	205
7 ―ラウリル 〃	152	200
8 ―ステアリル〃	124	215
9 ジ-オクチルホスフェート―ブチルアミン塩	118	205
10 ―オクチル 〃	104	210
11 ―ラウリル 〃	96	210
12 ―ステアリル〃	83	230
13 ジ-2-エチルヘキシルホスフェート―ブチルアミン塩	143	210
14 ―オクチル 〃	122	230
15 ―ラウリル 〃	111	230
16 ―ステアリル〃	85	240
17 ジ-ラウリルホスフェート―ブチルアミン塩	105	210
18 ―オクチル 〃	93	215
19 ―ラウリル 〃	87	235
20 ―ステアリル〃	76	240
21 ジ-オレイルホスフェート―ブチルアミン塩	75	220
22 ―オクチル 〃	67	225
23 ―ラウリル 〃	65	230
24 ―ステアリル〃	59	230

1 Test Sample
2 Total Acid Value
3 First Order pyrolytic temperature
4 Acidic phosphoric acid ester amin salt
5 di-butyl phosphate - butyl amin salt
6 - octyl "
7 - lauryl "
8 - stearyl "
9 di-octyl phosphate - butyl amin salt
10 - octyl "
11 - lauryl "
12 - stearyl "

TABLE 2 - The Properties of the Test Samples - Continued

13	di-2-ethyl hexyl phosphate	- butyl amin salt
14		- octyl "
15		- lauryl "
16		- stearyl "
17	di-lauryl phosphate	- butyl amin salt
18		- octyl "
19		- lauryl "
20		- stearyl "
21	di-oleyl phosphate	-butyl amin salt
22		- octyl "
23		- lauryl "
24		- stearyl "

TABLE 3 - Properties of Base-oil

表3 基油の性状

3 項目 \ 1 基油	2 150ニュートラルオイル
4 比重(15/4°C)	0.860
5 反応	14 中性
6 粘度(37.8°C, cSt)	32.29
(〃 , SUS)	151.5
(98.9°C, cSt)	5.352
(〃 , SUS)	43.79
7 粘度指数	109
8 流動点(°C)	−15.0
9 灰分(wt%)	0.000
10 全酸価(mgKOH/g)	0.00
11 アニリン点(°C)	107.0
12 硫黄分(wt%)	0.02
13 n-d-M 環分析:	
$\%C_A$	1.5
$\%C_N$	31.5
$\%C_P$	67.0

1 Base oil
2 150 neutral oil
3 Article
4 Gravity (15/4° C)
5 Reaction
6 Viscosity
7 Viscosity index
8 Flowage point
9 Ash content
10 Total phosphate value (mgKOH/g)
11 Anilin point (° C)
12 Sulfur content (wt%)
13 n-d-M ring analysis:
14 neutral

TABLE 4 - Sintering Power of Phosphoric Acid Ester Family

(base oil: initial load-sintering 45kg, load absorption 112kg)

表4 リン酸エステル類の耐焼付き性

(基油: 初期焼付き荷重45 kg, 融着荷重112 kg)

1 項目 \ 2 アルキル基	3 ブチル	4 オクチル	5 2-エチルヘキシル	6 ラウリル	7 オレイル
8 初期焼付き荷重(kg)					
9 中性リン酸エステル	71 (79)	79 (79)	56 (63)	71 (89)	50 (63)
10 酸性リン酸エステル	126 (126)	112 (112)	89 (89)	79 (100)	89 (89)
11 融着荷重(kg)					
12 中性リン酸エステル	126 (126)	126 (141)	126 (126)	126 (126)	141 (141)
13 酸性リン酸エステル	141 (158)	141 (141)	141 (158)	126 (126)	141 (158)

14 (注) 上段数値: P.0.10 wt%の場合, 下段( )内数値: P.0.20 wt%の場合

1 Article
2 Alkyl-group
3 butyl
4 octyl
5 2-ethyl hexyl
6 lauryl
7 oleyl
8 initial load-sintering (kg)
9 neutral phosphoric acid ester
10 acidic phosphoric acid ester
11 load-absorption (kg)
12 neutral phosphoric acid ester
13 acidic phosphoric acid ester
14 (Note): top figure: in case of P. 0.10wt%
bottom figure: in case of P. 0.20wt%

TABLE 5 - Sintering Power of Acidic Phosphoric Acid Ester Amin Salt (base oil: initial load sintering 45kg, load absorption 112kg)

表5 酸性リン酸エステルアミン塩の耐焼付き性

(基油：初期焼付き荷重45kg，融着荷重112kg)

2 アルキル基(酸性リン酸エステル) / 1 項目	3 ブチル	4 オクチル	5 2-エチルヘキシル	6 ラウリル	7 オレイル
8 初期焼付き荷重(kg)					
9 ブチルアミン塩	—	112 (112)	89 (89)	89 (89)	89 (89)
10 オクチル 〃	89 (89)	79 (100)	63 (71)	56 (71)	56 (63)
11 ラウリル 〃	79 (89)	71 (100)	63 (71)	56 (63)	56 (56)
12 ステアリル 〃	71 (79)	71 (89)	56 (63)	56 (63)	56 (56)
13 融着荷重(kg)					
14 ブチルアミン塩	—	141 (141)	141 (141)	126 (126)	126 (141)
15 オクチル 〃	141 (158)	141 (141)	141 (158)	126 (141)	141 (141)
16 ラウリル 〃	141 (158)	141 (141)	141 (158)	126 (141)	126 (141)
17 ステアリル 〃	112 (158)	126 (126)	141 (158)	126 (126)	126 (141)

18 注）上段数値：P.0.10 wt%の場合，下段（ ）内数値：P.0.20 wt%の場合

1 Article
2 Alkyl group (acidic phosphoric acid ester)
3 butyl
4 octyl
5 2-ethyl hexyl
6 lauryl
7 oleyl
8 initial load sintering (kg)
9 butyl amin salt
10 octyl "
11 lauryl "
12 stearyl "
13 load absorption (kg)

TABLE 5 - Sintering Power of Acidic Phosphoric Acid Ester Amin Salt - Continued

14 butyl amin salt

15 octyl "

16 lauryl "

17 stearyl "

18 (Note) top figure: in case of P. 0.10wt%
bottom figure: in case of P. 0.20wt%

TABLE 6 - Chemical Absorption of Phosphoric Acid Ester

Amount of chemical absorption: P 10^{-4}wt%

Condition: test sample oil (P 0.10wt%) 100g, deoxidized iron powder 20g, 170°C x 30min.

表6　リン酸エステル類の化学吸着性

化学吸着量: P 10^{-4} wt%

条件: 試料油(P 0.10 wt%)100 g, 還元鉄粉 20 g, 170°C×30 min

2 アルキル基 / 1 項目	3 ブチル	4 オクチル	5 2-エチルヘキシル	6 ラウリル	7 オレイル
8 中性リン酸エステル	5	4	3	5	4
9 酸性リン酸エステル	175	43	22	19	22
10 酸性リン酸エステルアミン塩					
11 ブチルアミン塩	—	21	18	27	23
12 オクチルアミン塩	135	22	25	30	22
13 ラウリルアミン塩	94	21	21	32	26
14 ステアリルアミン塩	96	28	36	31	25

1 Article
2 Alkyl-group
3 butyl
4 octyl
5 2-ethyl hexyl
6 lauryl
7 oleyl
8 neutral phosphoric acid ester
9 acidic phosphoric ester
10 acidic phosphoric acid ester amin salt
11 butyl amin salt
12 octyl "
13 lauryl "
14 stearyl "

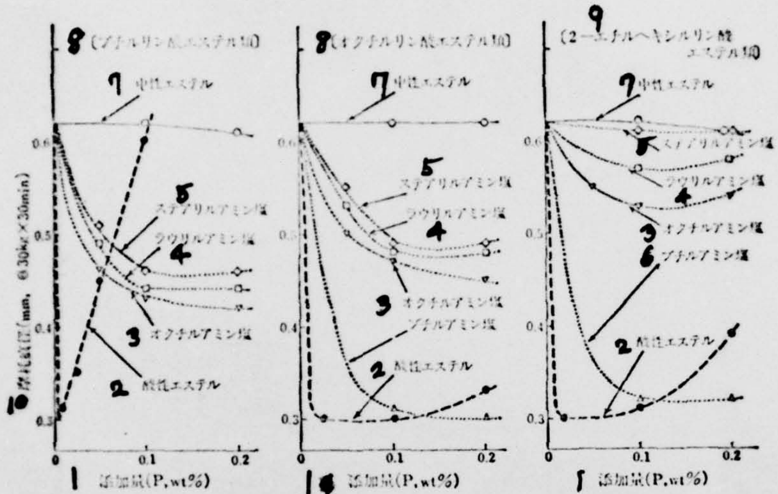


図1 ブチル, オクチル, 2-エチルヘキシルリン酸エステル類の摩耗防止性

Diagram 1 - Abrasion-Resistance Capacity of butyl, octyl, 2-ethyl hexyl phosphoric acid ester family

1 Amount of additive
2 Acidic ester
3 Octyl amin salt
4 Lauryl amin salt
5 Stearyl amin salt
6 Butyl amin salt
7 Neutral ester
8 (butyl phosphoric acid ester family)
9 (2-ethyl hexyl phosphoric acid ester family)
10 Traces of abrasion

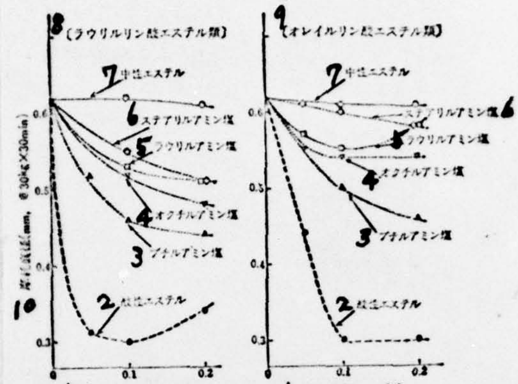
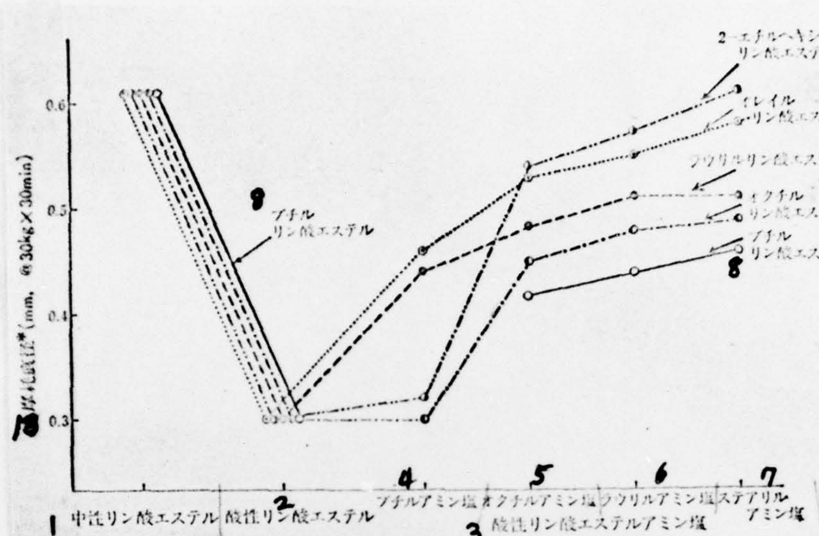


図2 ラウリル，オレイルリン酸エステル類の摩耗防止性

Daigram 2 - Abrasion-Resistance Capacity of Lauryl, Oleyl Phosphoric Acid Ester Family

1 Amount of additive
2 Acidic ester
3 Butyl amin salt
4 Octyl amin salt
5 Lauryl amin salt
6 Stearyl amin salt
7 Neutral ester
8 (Lauryl phosphoric acid ester family)
9 (Oleyl phosphoric acid ester family)
10 Traces of abrasion



* *リン分として0.20wt%までの添加濃度範囲で得られる最少摩耗痕径

図3 リン酸エステル類の摩耗防止性

Diagram 3 - Abrasion-Resistance Capacity of Phosphoric Acid Ester Family

* Minimum traces of abrasion obtained within the range of additive concentration (phosphate content 0.20wt%)

1 Neutral phosphoric acid ester
2 Acidic phosphoric acid ester
3 Acidic phosphoric acid ester amin salt
4 Butyl amin salt
5 Octyl amin salt
6 Lauryl amin salt
7 Stearyl amin salt
8 Butyl phosphoric acid ester
9 Octyl phosphoric acid ester
10 Lauryl phosphoric acid ester
11 Oleyl phosphoric acid ester
12 2-ethyl hexyl phosphoric acid ester
13 Trases of abrasion

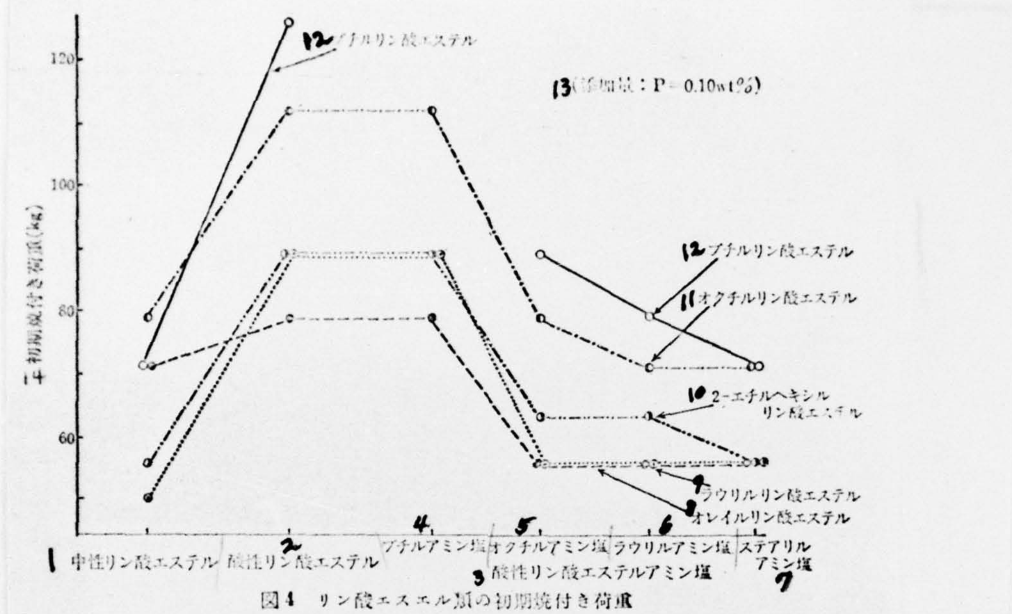


Diagram 4 - Initial Load-Sintering of Phosphoric Acid Ester Family

1 Neutral phosphoric acid ester
2 Acidic phosphoric acid ester
3 Acidic phosphoric acid ester amin salt
4 Butyl amin salt
5 Octyl amin salt
6 Lauryl amin salt
7 Stearyl amin salt
8 Oleyl phosphoric acid ester
9 Lauryl phosphoric acid ester
10 2-ethyl hexyl phosphoric acid ester
11 Octyl phosphoric acid ester
12 Butyl phosphoric acid ester
13 Amount of additive
14 Initial load sintering

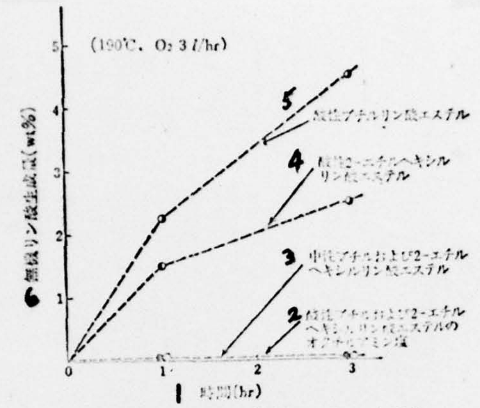


図5 熱分解による無機リン酸生成傾向

Diagram 5 - Formation of Inorganic Phosphoric Acid by Pyrolysis

1 Time

2 Octyl amin salt of acidic butyl and 2-ethyl hexyl phosphoric acid ester

3 Neutral butyl and 2-ethyl hexyl phosphoric acid ester

4 Acidic 2-ethyl hexyl phosphoric acid ester

5 Acidic butyl phosphoric acid ester

6 Amount of the formed inorganic phosphoric acid

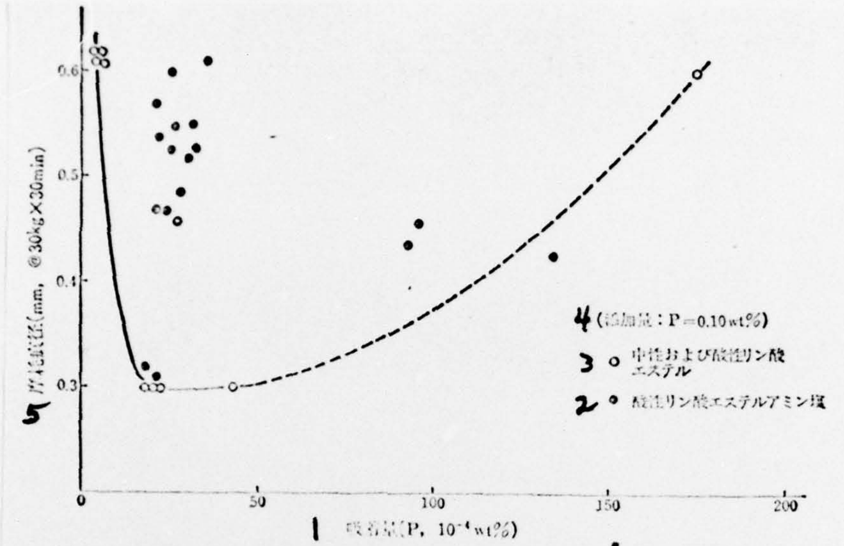


図6 化学吸着性と摩耗防止性

Diagram 6 - Chemical Absorption and Abrasion-Resistance Capacity

1 Amount of absorption
2 Acidic phosphoric acid ester amin salt
3 Neutral and acidic phosphoric acid ester
4 Amount of additive
5 Traces of abrasion

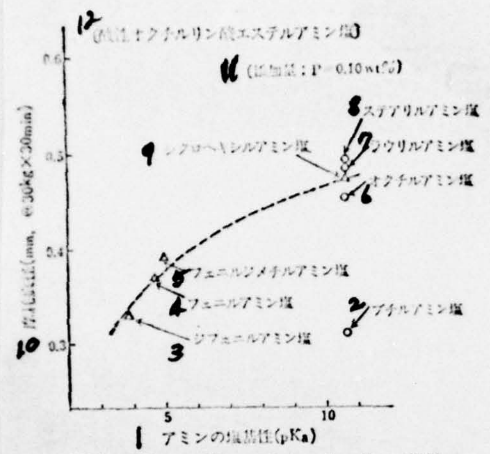


図7 酸性リン酸エステルアミン塩の摩耗防止性とアミンの塩基性

Diagram 7 - Amin salt-base and abrasion-resistance capacity of acidic phosphoric acid ester amin salt

1 Base of amin salt
2 Butyl amin salt
3 Di-phenyl amin salt
4 Phenyl amin salt
5 Phenyl di-methyl amin salt
6 Octyl amin salt
7 Lauryl amin salt
8 Stearyl amin salt
9 Cyclo hexyl amin salt
10 Traces of abrasion
11 Amount of additive
12 (Acidic octyl phosphoric acid ester amin salt)

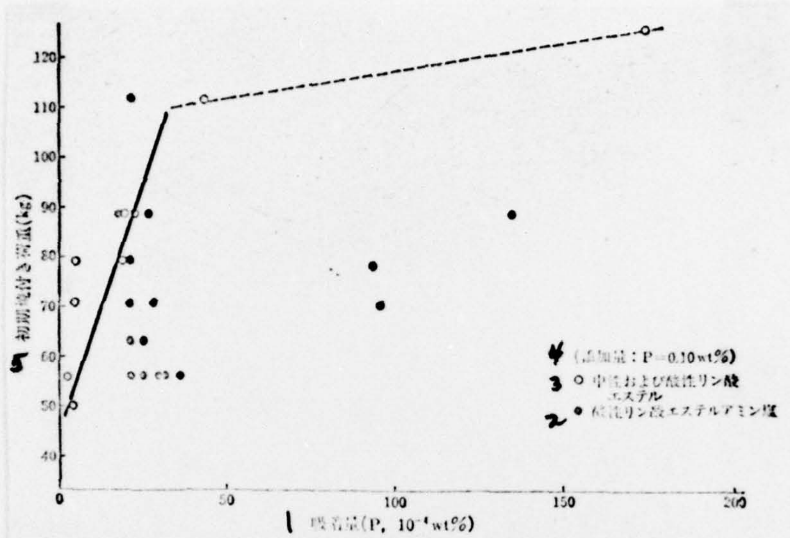


図8 化学吸着性と初期焼付き荷重

Diagram 8 - Chemical Absorption and Initial Load Sintering

1 Amount of absorption
2 Acidic phosphoric acid ester amin salt
3 Neutral and acidic phosphoric acid ester
4 Amount of additive
5 Initial load sintering

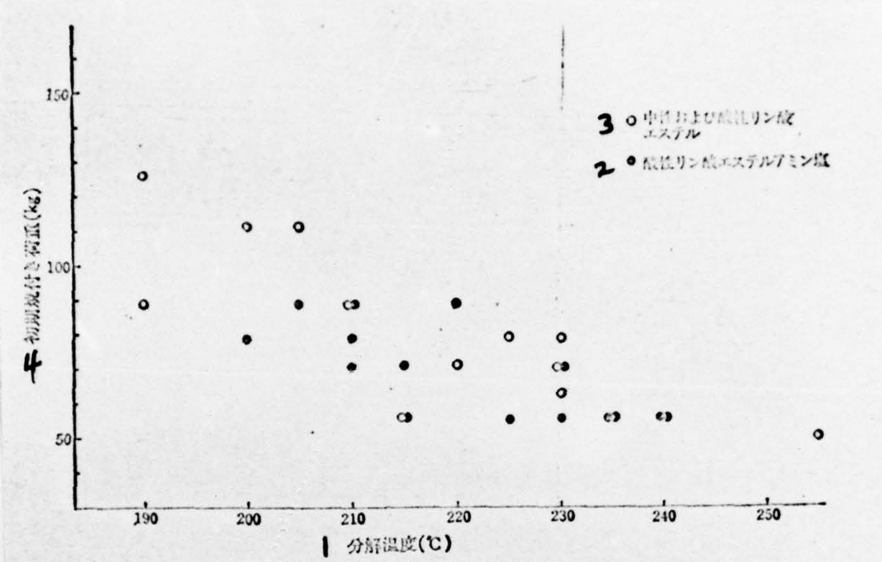


図9 熱分解温度と初期焼付き荷重

Diagram 9 - Pyrolytic Temperature and Initial Load Sintering

1 Pyrolytic temperature
2 Acidic phosphoric acid ester amin salt
3 Neutral and acidic phosphoric acid ester
4 Initial load sintering

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