Lubricants for HFC Refrigerant Compressors

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(Received September 6, 1993)

To arrest the depletion of ozone layer, CFC-12, which has been widely used in automobile air conditioners or household refrigerators, is being replaced by an alternative refrigerant HFC-134a. At this juncture, it is not possible to maintain the same level of reliability and refrigeration performance when the CFC refrigerant alone is replaced by HFC-134a. A new refrigerant requires a new system and other materials to complement it, and the development of those materials is urgently needed. Among those materials, the development of suitable refrigeration compressor lubricant is of utmost importance in securing good performance, since the lubricant characteristics directly influence the durability of compressors. This paper examines refrigeration lubricants, polyol esters and polyalkylene glycols as to their miscibility with HFC-134a, lubricity and chemical stability, since those properties are indispensable to lubricants used in any kind of refrigerant compressors. Among those, miscibility is determined solely by the molecular structures, while lubricity and chemical stability can be improved mainly by additives. It is possible to give the lubricant well-balanced properties by modifying chemical structures of base oils and by mixing various kinds of additives. There still remain some problems to be solved, however, before the reliability of refrigerating system is enhanced even more, including: (a) finding the rate of additive consumption, (b) examining the interaction among additives, (c) investigating the effects of the interaction among additives on other properties such as chemical stability, lubricity and others. Moreover, it should be taken into account that no matter how excellent the lubricity of the lubricant be, lubricity drops sharply as refrigerant dilution ratio increases.

1. Introduction

CFCs have been widely used as excellent refrigerants since the 1930s. Nowadays, however, their use is regulated to arrest the depletion of the ozone layer. In November 1992, in Copenhagen, Denmark, the international Montreal Protocol treaty was amended to accelerate the phase out of CFCs, to be completed by January, 1996. The parties also came to an agreement to restrict the use of HCFCs and to completely phase it out by 2030. Now, new technology for using alternative refrigerants is urgently needed.

As an alternative for CFC-12, which refrigerant has been widely used for automobile air conditioners or domestic refrigerators, HFC-134a is regarded as an appropriate refrigerant. The main reasons for its choice follows: similarity of its thermodynamic properties to those of CFC-12, no ODP (Ozone Depletion Potential), and its non-toxicity as judged by PAFT (Program for Alternative Fluorocarbon Toxicity Testing)3).

At this juncture, it is not possible to maintain the same level of reliability and refrigeration performance when the CFC refrigerant alone is replaced by HFC-134a. A new refrigerant requires a new system and other materials to complement it. Thus the change of refrigerant brings about problems of modifying the system itself, and finding or developing materials which are compatible with the alternative refrigerant HFC-134a, e.g., refrigeration compressor lubricants, desiccants, electronic insulating films and magnet wires for household refrigerators, hoses and gaskets for automobile air conditioners and so on, depending on the kind of machines in which the refrigerant is used. Among those materials, the development of suitable lubricant is of utmost importance in securing good performance, since lubricant characteristics directly relate the durability of the compressor, the component which could be called the heart of refrigerating or air conditioning systems.

In this study, miscibility of lubricant with HFC-134a, lubricity, chemical stability and others, which are indispensable to any lubricant for all kinds of refrigerant compressors, were examined.

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2. Required Properties of Refrigerator Oil

Miscibility, lubricity, and chemical stability are commonly required properties of any refrigerator oil. Other properties are also required depending on their uses. For example, in household refrigerators, lubricant must have high electrical insulating properties, since refrigerators have hermetic units where electronic components are directly in contact with oil or immersed in it. In case insulating properties are low, electric current would stray off.

There is also a problem of lubricant compatibility with other materials, that is, compatibility with insulating films and magnet wires used in household refrigerators, compatibility with materials used in hoses and gaskets found in automobile air conditioners, and compatibility with desiccating agents, because lubricant entraining refrigerant circulates in the system. Incompatibility will result in the deterioration of lubricant and materials coming in contact with it. Compatibility, however, cannot be determined by lubricant properties alone. It depends on the interaction of the lubricant with other materials. Examination of compatibility involves a large number of materials whose compositions are not always clear to users, and it is extremely difficult to discuss the matter of compatibility within a limited space. This paper, therefore, deals only with such properties of the lubricant as miscibility, lubricity, chemical stability, and insulating property.

3. Test Procedure

3.1. Evaluation of Miscibility

Miscibility was evaluated in accordance with the method prescribed in JIS K 2210 (equivalent to ASHRAE Standard). In the case of HFC/lubricant mixtures, immiscible zones exist both in high and low temperature zones, as shown in Fig. 1. The two points in Fig. 1, namely the UCST (Uppermost Critical Solubility Temperature) and LCST (Lowest Critical Solubility Temperature), mark the upper and lower limits of the miscible temperatures. This paper deals only with the UCST influences the performance of compressor. The lower the UCST, the better the miscibility.

3.2. Evaluation of Lubricity

3.2.1. Lubricity in the Atmosphere

First, the lubricity in the atmosphere was examined with Falex type friction tester, which is widely used for evaluation of lubricity of refrigerator oil. The journal was rotated at a speed of 290 min⁻¹ linearly with two V-blocks contacting the journal. After rotating for five minutes at an initial load of 0.2 kN, the applied load was increased by 0.2 kN/min until the friction coefficient sharply increased as a result of seizure. Lubricity was evaluated by the seizure load at room temperature.

3.2.2. Lubricity of Refrigerant/Lubricant Mixture

Lubricity in an environment influenced by a refrigerant is entirely different from that in the air. Refrigerant effect on lubricity must be taken into account since the friction occurs in an environment influenced by refrigerant. The apparatus with a pressurized container was set up for evaluation of lubricity of refrigerant/lubricant mixture. Shown in Fig. 2 is a schematic diagram of the tester. Various modes of friction can be chosen by changing the form of the sliding part.
Here, the sliding mode, shown in the figure, was adopted.

The pressurized container was rinsed with toluene before the tests. After the container was dried and degassed, the refrigerant/lubricant mixture was introduced into the container. The friction test was carried out by rotating one slider against another mounted in the container, at a speed of 1,200 min⁻¹. After rotating for five minutes, at an initial load of 0.2 kN, the applied load was increased at an increment of 0.2 kN/min. The load was applied until the friction coefficient sharply increased as a result of seizure. Lubricity was evaluated by the seizure load at room temperature.

3.3. Evaluation of Chemical Stability
Chemical stability was evaluated in accordance with the method prescribed in JIS K 2210 (equivalent to ASHRAE Standard).

3.4. Evaluation of Electrical Insulating Property
Insulating property was evaluated in terms of volume resistivity in accordance with the testing method of electrical insulating oils, prescribed in JIS C 2101.

3.5. Prepared Samples
At present polyol esters (POEs) and polyalkylene glycols (PAGs) are attracting attention as promising base oils for HFC-134a refrigerator lubricant, mainly because of their good miscibility with HFC-134a and their availability. The former is becoming to be used in household refrigerators, and the latter, in automobile air conditioners. Although it has been reported that other base oils such as carbonates⁶, fluorosilicons⁷ etc. have good miscibility with HFC-134a, this study deals with POE and PAG only, because other base oils have not been yet examined thoroughly. Shown in Tables 1 and 2 are the POEs, PAGs, respectively, which were taken as test samples. Alkylbenzene and mineral oils are listed for reference in Table 3, for the sake of comparing the POEs and PAGs with conventional lubricants used in household refrigerators and automobile air conditioners.

4. Results and Discussions

4.1. Miscibility
As pointed out earlier, there are several properties which are indispensable to refrigerator oils. Although some of them can be enhanced by additives, but not miscibility. Miscibility is dependent on bonding between refrigerant and lubricant by hydrogen. Hence, this property is affected by polar groups introduced into the lubricant molecule, and their structural configurations. Inoue et al.⁸ explained the miscibility between HFC-134a and POEs by three dimensional solubility parameters and ¹H-NMR spectral analyses.

4.1.1. Miscibility of HFC-134a/POE Mixture

<table>
<thead>
<tr>
<th>Table 1 Prepared Polyol Esters</th>
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NPG: Neopentyl glycol; TMP: Trimethylol propane; PE: Pentaerythritol.

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<th>Table 2 Prepared Polyalkylene Glycol</th>
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<td>PAG-2</td>
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<td>PAG-3</td>
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<td>PAG-4</td>
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<td>PAG-5</td>
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PAG: Polyalkylene glycol.

Fig. 3 Miscibility Curve for Mixtures of HFC-134a and TMP Ester

Table 3 Usual Lubricants Prepared for Reference

<table>
<thead>
<tr>
<th>Lubricant</th>
<th>Viscosity at 40°C [mm²/s]</th>
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<tr>
<td>Alkylbenzene</td>
<td>61.0</td>
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<tr>
<td>Naphthenic mineral oil-A</td>
<td>55.5</td>
</tr>
<tr>
<td>Naphthenic mineral oil-B</td>
<td>111.0</td>
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石油学会誌 Sekiyu Gakkaishi, Vol. 37, No. 3, 1994
Esters are formed by reaction of alcohols and fatty acids. There are many variations according to the combinations of the two. In this section, miscibility, in terms of UCST, is discussed from the viewpoint of molecular structure of the lubricants.

(a) Dependency of miscibility on molecular structure of fatty acid

Shown in Figs. 3 and 4 are the miscibility of trimethylol and pentaerithritol esters with HFC-134a. The molecular structures of the fatty acids, which were used as a starting material for synthesis of esters, had considerable influence on the miscibility. Esters with a branched carbon chain had better miscibility than those with a straight carbon chain.

Fatty acids were separately tested to examine the effect of molecular structure of fatty acids on miscibility. Shown in Fig. 5 are miscibility of two fatty acids (n-Cs and br-Cs acids) with HFC-134a. The fatty acids were miscible with HFC-134a and their miscibility were greatly dependent on their molecular structures. Here again, the fatty acid with a branched carbon chain had better miscibility than that with a straight chain.

It was found that the structure of fatty acid is indirectly related with miscibility. Miscibility is dependent on the hydrogen bonding which occurs at carboxyl group in fatty acid. It is considered, therefore, that the effect of carboxyl group is ascribable to in ester, although fatty acid itself changes its structure upon esterification.

(b) Dependency of miscibility on carbon number of fatty acid

The relationships between number of carbons of

Fig. 4 Miscibility Curve for Mixtures of HFC-134a and PE Ester

Fig. 5 Miscibility Curve for Mixtures of HFC-134a and Fatty Acid

Fig. 6 UCST vs. Carbon Number of Fatty Acid with Branched Carbon Chain

Fig. 7 UCST vs. Carbon Number of Fatty Acid with Straight Carbon Chain
fatty acids and the UCST are shown in Figs. 6 and 7 for three polyol esters: pentaerithritol (PE), trimethylol propane (TMP), and neopentyl glycol (NPG) esters. The greater the carbon number of fatty acids used in esterification, the lower was the miscibility of the ester. Among the three, the ester of NPG had the best miscibility, and the ester PE, the worst.

While miscibility improved with decrease in the number of carbons of the fatty acids, viscosity decreased, as shown in Figs. 8 and 9. Generally speaking, the viscosity grade at 40°C is 20—50 mm²/s in household refrigerators and 50—120 mm²/s in automobile air conditioners. Further, refrigerator oils must have UCST of lower than −20°C in household refrigerators, and lower than −10°C in automobile air conditioners. It is not practical satisfy both properties, that is, miscibility and optimum viscosity grade, at the same time, only by changing the number of carbons of the fatty acid. On the other hand, it is difficult to use another polyol alternatively, because only a few practical polyols are available. In order to balance these contradicting characteristics, molecular structures of esters are designed by skillfully combining several kinds of fatty acids, which must include at least one fatty acid with a branched carbon chain. It seems, that polyols are sometimes mixtures of two polyols, but detailed information is kept confidential by suppliers and not provided to users. Listed in Table 4 are some commercial lubricants together with their viscosity and UCST. All polyol esters prepared had LCSTs higher than 60°C. The best balance was obtained by mixing fatty acids with carbon numbers of approximately from 6 to 10.

### 4.1.2. Miscibility of HFC-134a/PAG Mixture

Table 5 shows the miscibility of mixture of PAGs with HFC-134a. PAGs, as well as POEs, showed miscibility with HFC-134a, depending on their molecular structures and viscosities. The samples had their molecular structures modified in order to satisfy various properties. It was possible to balance the miscibility with HFC-134a and viscosity, as far as the prepared samples were concerned.

### 4.2. Lubricity

CFC–12 itself has high lubricity, and works well as EP (Extreme Pressure) agent in CFC–12/lubricant mixtures. Although it has been reported that HFC-134a, too has lubricity by itself, its EP effect is very low, compared with CFC–12. Lubricants of higher lubricity, therefore, are required when HFC-134a is used, rather than when CFC–12 is used. For this purpose, i.e. to improve the lubricity, a phosphorus type EP agent was added to the lubricants and tests were carried out to evaluate the additive effect.

#### 4.2.1. Lubricity in the Atmosphere

First, lubricity in the atmosphere was evaluated by Falex type tester. For POE and PAG, as shown in Figs. 10 and 11, the friction coefficients are plotted against applied load, with additive content as a parameter. POE and PAG did not show...
sensitivity to additives as much as did conventional lubricants. Additive showed no effect before 0.3 wt%, while with mineral oil and alkylbenzene, the additive showed effect from 0.1 wt%. This can be attributed to the strong adsorption of POE or PAG to metal surface, which prevents reaction between the EP agent and the metal\textsuperscript{13}. Another explanation is that the EP agent is highly soluble in POE and PAG, which keeps the concentration of EP agent low near the metal surface, and subsequently weakens the reaction between the EP agent and the metal. The friction coefficient, however, was kept low even at high loads when 1.0 wt% EP agent was added to the base oil in POE and 0.3 wt% to PAG. It is possible, therefore, to improve lubricity by additives.

4.2.2. Lubricity of CFC-12/Lubricant Mixture

Shown in Fig. 12 are plots of the relationship between applied load and friction coefficient as a function of refrigerant ratio of CFC-12/alkylbenzene mixture. The friction coefficient sharply increased at a certain load due to seizure. The seizure load was highest in pure CFC-12 liquid and lowest in pure lubricant. The same tendency was observed with the combination of CFC-12/mineral oil A (and B).

4.2.3. Lubricity of HFC-134a/Lubricant Mixture

Shown in Fig. 13 are plots of the relationship between applied load and friction coefficient for HFC-134a and POE mixture. The results for TMP ester A containing 0.4 wt% EP agent is shown as an example. The seizure load was the lowest in pure HFC-134a and became lower with increase in HFC-134a content, since there is very little lubricity-improving property in HFC-134a itself.

4.2.4. Relation between Seizure Loads of Lubricant in the Atmosphere and in Mixture with Refrigerant

The seizure loads of the lubricants mixed with the refrigerant are plotted against those in the atmosphere (Falex-type friction tester) in Fig. 14. The lubricants which had high seizure loads in the atmosphere tended to have higher seizure loads also in refrigerant influenced environment. Additives also had similar tendency of effectiveness in refrigerant influenced environment. When appropriate additives were present in suitable...
amounts in the base oil, the combination of HFC-134a/POE or HFC-134a/PAG showed the same or a higher level of lubricity as those of CFC-12/alkylbenzene or CFC-12/mineral oil A mixture.

It has been reported that phosphorus EP agent is effective for improving lubricity only in the atmosphere, but not in vacuum\(^{15}\). However, the EP agent was effective here because it was impossible to completely displace the atmosphere by the refrigerant, and some air remained in the apparatus. In automobile air conditioners or household refrigerators, residual air is sure to remain in the system and can promote function of the EP agent.

4.2.5. Effect of Refrigerant Mixture Ratio on Seizure Load

Shown in Fig. 15 are the relationship between seizure load and refrigerant mixture ratio of HFC-134a/POE and CFC-12/alkylbenzene. As the refrigerant content increased, metal contact of sliding parts was likely to occur because of lowered viscosity and subsequent decrease in lubricating film thickness.

With CFC-12, seizure loads increased with increase in refrigerant content, since the refrigerant itself has lubricating property, while functions as an EP agent. By contrast, HFC-134a is void of lubricity and lubricity of the mixture sharply dropped when the refrigerant ratio exceeded above 50% in volume. These results indicate that wear of sliding parts would accelerate with increase in the refrigerant ratio, as ascertained by experiments\(^{10}\). This behavior was common when HFCs were used as refrigerant.

Accordingly, it is important to keep the lubricant content in sliding parts over 50% in HFC-134a compressors, by means of modification of compressor design or improvement of lubricant miscibility and so on.

4.3. Chemical Stability

The stability of refrigerator lubricants has been examined in condition of mixture with refrigerant, since lubricants are always used in refrigerant influenced environment. Unlike CFC-12 or HCFC-22, HFC-134a does not affect the stability of lubricant, because HFC-134a itself is chemically stable compared with those containing element of chlorine in the molecule. Lubricants for HFC refrigerant compressors, however, have polar groups in the molecule to make them miscible with HFC refrigerant. This makes lubricant itself unstable. In this section, the problems of stability peculiar to POE and PAG will be examined.

4.3.1. Chemical Stability of POE

One of the biggest problems with ester oil is that it is vulnerable to hydrolysis. Still worse, its affinity with moisture is stronger than that with mineral oil or alkylbenzene. The chemical structure of ester makes it hardly possible to avoid hydrolysis completely, although it can be prevented to a certain extent by modifying its molecular structure. These characteristics of esters can cause some problems when moisture enters the lubricant. For example, the fatty acid produced by hydrolysis may cause clogging of the capillary tube by formation of metal soap or may cause abnormal wear of moving parts by copper plating in the household refrigerators. Excessive formation of fatty acid might cause corrosive wear of sliding parts or may harm stability of motor materials in household refrigerators.

Shown in Fig. 16 are relationship between water content and acid value in a sealed tube test. The increase in acid value indicated the formation of fatty acid by hydrolysis. This harmful fatty acid can be deactivated by epoxide additives, which are known as deactivators. The epoxide compound reacts with the fatty acid and neutralizes it. The effect of an epoxide is shown in the same figure. Fortunately, household refrigerators employ...
watertight hermetic units, unlike automobile air conditioners. Here, there is very little chance of moisture entering from the outside once the refrigerator is assembled; but moisture must be controlled before and during assembly.

4.3.2. Chemical Stability of PAG

PAG has no problem concerning hydrolysis except the case in which molecular structure is modified by esterification. PAG, however, is vulnerable to degradation when combined with chlorides or exposed to oxidation in the atmosphere.

First, the harmful effect of chloride is examined. It is quite possible that a small amount of CFC-12 may remain in compressors in use when CFC-12 is replaced by HFC-134a. Further, a small amount of chloride may remain in the system when compressor parts are rinsed with chlorinated organic solvent such as CFC-113 or trichloroethylene. Thus, it was necessary to examine the effect of CFC-12 or CFC-113 on the stability of PAG.

Sealed tube tests indicated that chlorides have harmful effects on PAG stability, as shown in Fig. 17. While HFC-134a showed very little change of color, CFC-12 and CFC-113 showed a great change of color, which indicated that CFC-12 and CFC-113 deteriorated the lubricant stability to a great extent. CFC-12, therefore, must be completely removed, when former CFC-12 refrigerant compressors are remodelled by exchanging used CFC-12/mineral system with HFC-134a/PAG system. There is, however, an example of CFC-12/PAG combination system being applied to automobile air conditioners by stabilizing PAG with additives. It is presumed that harmful species (like HCl) produced by decomposition of CFC-12 itself or reaction between CFC-12 and lubricant are deactivated by additives such as epoxy or phosphate compounds. The additive effect of a phosphate is shown in the same figure. Sooner or later, we will be relieved of CFC related problems, because CFCs are to be phased out in the future.

Next, problem of oxidation is discussed. When a small amount of residual air is in the system, the lubricant becomes oxidized. Oxidation causes oil molecule to degrade into lower molecular materials, which are easily volatilized, with a subsequent loss of weight. Synthetic oils like PAGs show a clear relationship between the extent of oxidation and loss of weight, because most of their oxidation products volatilizes, leaving little amounts of solid-like materials behind. Thus, oxidation stability was evaluated by loss of weight when lubricant in a beaker was heated in an oven at 150°C. The results are shown in Fig. 18. The tests showed that PAG was susceptible to oxidation, and that the loss in weight increased sharply with length of heating. Addition of DBPC (di-tert-butylparacresol, 0.5% in weight) as an oxidation inhibitor stabilized the PAG.
Oxidation inhibitors, therefore, are indispensable to HFC-134a compressor lubricants which are PAG-based.

4.4. Electrical Insulating Property

In household refrigerators the electrical insulating property of lubricants is as important as miscibility and lubricity, since electronic components are directly in contact with oil. If the insulating property is low, electricity can stray off. Shown in Fig. 19 are insulating properties of some base oils in terms of volume resistivity. Volume resistivity of PAG and ester oil were lower those of mineral oil and alkylbenzene. The former two had greater affinity for moisture, and volume resistivity decreased sharply with increased moisture content. Especially in the case of PAG, its initial low insulating property was lowered even further by its stronger affinity for moisture. Although insulating of ester oil is lower than that of mineral oil, it is considered to be allowable for practical use. Moisture, however, showed to be controlled with great care before and during assembly.

5. Conclusion

Miscibility, lubricity and chemical stability are commonly essential properties of any refrigerator lubricant. Other specific properties are additionally required depending on their use. For example, in household refrigerators, lubricant must have high electrical insulating property. Among the various properties, miscibility and electrical insulating properties are determined based on molecular structure of base oil itself, while lubricity and chemical stability can be improved by additives.

When CFC–12 or HCFC–22 are used as a refrigerant, refrigerating systems need no or perhaps very few additives. In the systems using HFC–134a, however, it is a key technology to furnish with well-balanced properties to the base oil, by the use of various kinds of additives, as well as by modifying chemical structure of base oil.

Presently, PAG-based lubricants have come to be used in automobile air conditioners, while POE-based ones are used in household refrigerators, by skillful modification of molecular structure of base oil and by combination of various additives. When it comes to practical application, however, there still remain some points yet to be examined in order to enhance the reliability of refrigerating system for long-range operation: (a) the durability of additives, (b) the chemical interactions among additives, (c) the effect of their interaction to other properties such as chemical stability and lubricity. At the same time, it is none the less important to design the refrigerant compressor system by taking into account the characteristics of the lubricant. No matter how excellent the lubricity of the lubricant itself, the lubricity sharply drops with excessive increase in refrigerant mixture ratio in HFC–134a compressors.

References


Fig. 19 Insulating Property of Base Oils
オゾン層の破壊を防止するために、カーフォンや家庭用冷蔵庫に広く用いられてきた CFC-12 は HFC-134a に替わりつつある。しかし、これらの機器に対して、単に CFC 系冷媒を HFC-134a に替えただけでは従来と同じ信頼性は得られない。新しい冷媒を用いた場合、新しいシステムとして用いられる新たな材料の開発が必要である。材料の中で、潤滑油の特性は圧縮機の耐久性に直接影響するので、適正な潤滑油の選定は極めて重要である。本報では、冷媒圧縮機用潤滑剤に対して要求される特性、冷媒との相溶性、潤滑性、化学安定性などについてポリアルキレングリコール油とポリオールエステル油を評価した。潤滑油の分子構造の変化と多くの添加剤の適用によってパラメータの取れた潤滑剤が得られた。しかし、冷凍システムの信頼性をさらに高めるためには、次の課題を解決しなければならない。(a)添加剤の消耗速度の低減、(b)添加剤間の相互作用、(c)添加剤の相互作用の化学安定性、潤滑性等への影響。さらに、潤滑剤がどんなに潤滑性に優れていても、冷媒の濃度が高くなると潤滑性は極端に低下することも考慮しなければならない。

Keywords
Synthetic lubricant, Tribology, Lubrication, Environment, Fluorocarbon, Alternative refrigerant

石油学会誌 Sekiyu Gakkaishi, Vol. 37, No. 3, 1994