技術解説

Polyalkylene Glycol (PAG) as Environmentally Acceptable Shock Absorber Oil

環境対応型ショックアブソーバ油としてのポリアルキレングリコール（PAG）

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要　　旨

近年の環境負荷低減を目的とした欧州REACH規制や米国Vessel General Permit等の強化に伴い，環境対応技術はより広い領域の分野で求められている．KYBにおいては多くの製品の作動流体として油圧作動油を用いており，油圧作動油に対しても環境負荷低減が求められるようになっている．このためKYBにおいても，将来想定される環境対応要求に対し準備する必要性がある．

本報では，環境対応技術として作動油に求められる要件，及び環境対応型作動油（Environmentally Acceptable Lubricants : EAL）であるポリアルキレングリコール（Polyalkylene Glycol : PAG）について解説する．また，PAGを自動車用ショックアブソーバの動流体として適用した事例について紹介し，KYB製品における環境対応技術の可能性について述べる．

Abstract

Due to restrictions, such as European regulation EC/1907/2006 (REACH), substances for environmentally friendly and biodegradable lubricants getting more in focus of passenger car OEM's and suppliers R&D activities. Eco-tox properties are now mandatory in the U.S. Vessel General Permit. Goal of the present investigation is to characterize the tribological performance of different shock absorber fluids, including those as environmentally acceptable lubricants (EAL) declared. Therefore, four lubricants differing in their base oil were tested under laboratory conditions, in bench tests and finally in vehicle tests. The oils tested are composed of Group III-mineral base fluid and polyalkylene glycol (PAG) based oil, which is classified as readily biodegradable EAL. All oils are fully formulated with standard additives for shock absorbers. Friction properties under oscillating sliding conditions and Stribeck-curves were determined besides damping force in real applications. Subjective car evaluation and sensor-based objective data recording on public roads gave good conclusions how the oil and its tribological properties can influence the driving characteristics (vehicle stability and comfort).

1 INTRODUCTION

Biodegradability, toxicity, renewability and bioaccumulation are the key words which are closely linked to the environmental impact of lubricants such as engine oils and hydraulic fluids. These issues of bio-no-tox properties are getting more and more in focus of original equipment manufacturers OEM's in automotive, marine and hydraulics sectors. Over the last 30 years European, American and Asian governments released several regulations which address the toxicity of chemicals (1907/2007/EC REACH) or provide the legal basis for lubricants classifications (EPA Vessel General Permit) and so called Ecolabels (2005/360/EC). The European REACH regulation (Registration, Evaluation, Authorization and Restriction of Chemicals) communicate information on chemicals ensuring that manufacturers and customers are aware of health and safety characteristics of the products. Many countries worldwide offer eco-labels for several industrial product categories including lubricants and their additives. These labels provide a definition which product have a low environmental impact, do not affect aquatic organisms or other animals and furthermore consider the aspect of biodegradability and renewability. Unfortunately these legislation vary in a wide range from country to country and indicate a lot of "bio and eco" terms such as[1]:

- EAL for Environmentally Acceptable Lubricants
- ECL for Environmentally Considerable Lubricants
- EFL for Environmentally Friendly Lubricants
- Hydraulic fluids for environmental impact
- Biodegradable fire resistant hydraulics fluids
- Eco-evaluated/eco-friendly fluids
- Oil respectful of the environment
- Eco lubricants

In comparison to other existing national eco-labels, such as the Blue Angel (Germany), the Eco Mark (Japan) and the Nordic Swan (northern European countries), the EU Ecolabel intensified the criteria for an approval as environmental friendly lubricant. In order to get the approval by EU Ecolabel regulation, the lubricant has to fulfill several criterions. The following points listed below are valid for hydraulic fluids (listed as Category 1 lubricants)[2), 3)]:

- No R phrase triggering materials (Risk phrases)
- Cumulative mass percentage ≤0.1% of substances categorized 'very toxic (G)' with acute aquatic toxicity ≤ 1 mg/L
- Readily aerobic biodegradation > 90%
- No halogen or nitrite compounds or heavy metals
- Renewable carbon content > 50%
- Impact on CO_2 emission as outlined in the standard
- ISO 15380 standards for technical performance criteria

Environmentally acceptable lubricants are commonly classified according the type of base fluid used in the formulation. Beside vegetable oils, produced by use of canola, soybeans or sunflower crops, synthetic esters and polyalkylene glycols (PAG) represent the major share of these base fluids. Group I, II, III and IV base oils which are mainly related to mineral base oils cannot be classified as EAL or environmentally friendly lubricants due to their high toxicity against organisms, inherently low range of biodegradation or not given renewability. The United States Environmental Protection Agency (EPA) summarizes the major factors of different base oil types regarding their biodegradation, toxicity and bioaccumulation potential. Comparing these factors the majority of currently used lubricant base oils have a low grade of biodegradation, a high potential for bioaccumulation and a measurable toxicity towards marine organisms[4]. PAGs, esters, and vegetable oils are candidate base stock for lubricants with low environmental impact (see

Table 1 Comparative environmental behavior of lubricants base oil type. [4]

Base Oil	Base Oil Source	Biodegradation	Bioaccumulation	Toxicity
Mineral Oil	Petroleum	Persistent/Inherently	Yes	High
PAG	Petroleum – synthesized hydrocarbon	Readily	No	Low
Synthetic Ester	Biological sources	Readily	No	Low
Vegetable Oil	Naturally occuring vegetable oils	Readily	No	Low

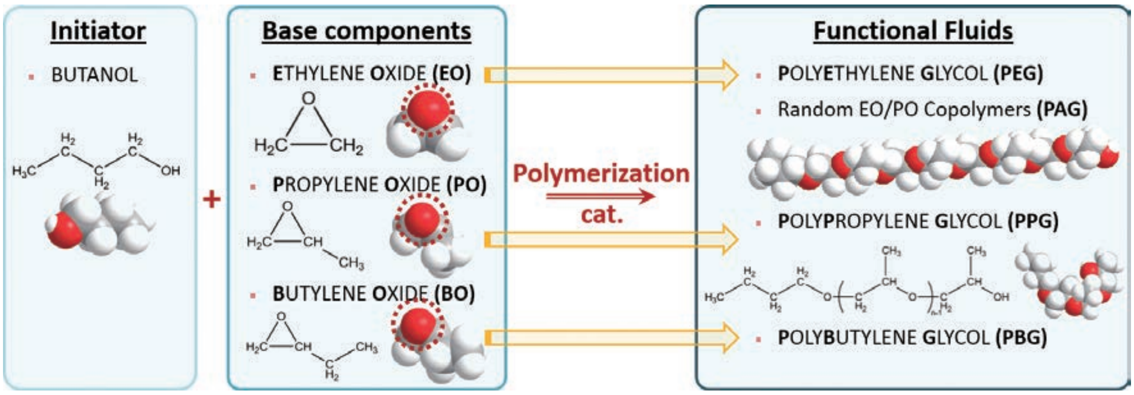

Fig. 1　Polymerization process of different polyalkylene glycol base oil types. Red colored atoms represent oxygen.

Table 1).

Polyalkylene glycols offer an attractive alternative to conventional hydraulic fluids due to several special oil characteristics described in the following context. The long history of PAGs goes back to the early 1940s where it was developed as one of the first synthetic base oils. The big advantage of PAGs is that they can be chemically designed to meet a wide range of performance characteristics in industrial applications. There is no other base available on the market which offers such versatility.

The manufacturing process of PAGs includes the reaction of an initiator (for example butanol) with one or more alkylene oxides (EO – ethylene oxide, PO – propylene oxide or BO – butylene oxide) under alkaline conditions and elevated temperatures (see Fig. 1). It is possible to add the oxides as single oxides forming a homopolymer such as polyethylene glycol (PEG) or as random oxides creating a copolymer of ethylene – and propylene oxide (EO/PO copolymer) for example. The choice of initiator and alkylene oxides and their different ways to react gives PAGs a wide range of intrinsic properties and makes the creation of tailor-made products possible. Additionally, PAGs have excellent lubricant film forming characteristics. The reason for this is the oxygen rich chemical structure of PAG (high polarity).

In comparison to PAG, mineral base oil, produced from crude oil or by synthetizing petroleum components, consists of a long chain (backbone) of hydrocarbon molecules without any oxygen. Every third atom along the backbone the PAG polymer is an oxygen atom (see Fig. 1) which results in a high affinity to metal surfaces. This feature contributes moreover mild antiwear and extreme pressure properties[5]. Typical oil characteristics of PAGs are:

- Wide viscosity range (ISO VG 7 to 10000)
- High viscosity index (up to 300)
- Water solubility and oil tolerance
- Hydrolytic stability
- Oxidative and thermal stability
- High flash point and low volatility
- Good additive compatibility
- Biodegradability and low toxicity

The high hydrolytic stability of PAGs makes them a good choice for industrial equipment used around waterways. This is caused by the ability to act as polymeric sponges binding the water, which entered the system, through hydrogen bonding and rendering it inert even at levels of up to 20,000 ppm (mineral base oil <500 ppm).

KYB is one of the world largest manufacturer of shock absorbers and hydraulic systems. Therefore a huge interest in improving these system is always given. By definition shock absorber are hydraulic or mechanical devices attached to car chassis and car body structure. The main function of the device is to damp the kinetic energy of road shocks, caused by road conditions or severe impacts, by converting it into another form of energy (heat)[6]. Beside a high level of comfort for driver and passengers as well as driving performance and stability, car shock absorbers make a significant contribution to driving safety (braking distance). A typical passenger car shock absorber undergoes 75 million shaft cycles in approximately 80,000 kilometers, which means 900 cycles per kilometer at various sliding speeds of piston and piston rod. Using the shock absorber longer than recommended by the passenger car OEM several problems could occur and influence the car security. Poor steering responsiveness, lack of control, noises and a significant increase in braking distance are the results of bad shock absorber performance related to mileage and wear reasons. Nowadays, a large number of shock absorber is designed as so called 'twin-tube' shock absorber, which means that it consists of two cylinders (working or inner cylinder and outer cylinder). Fig. 2 shows a sectional view of a typical passenger car twin-tube shock absorber including piston, bushing, and oil seal as main sliding parts.

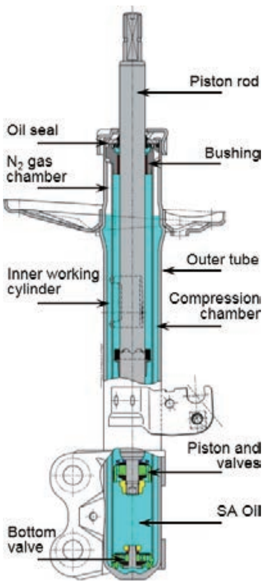

Fig. 2 Sectional view of a twin-tube shock absorber.

The outer cylinder serves as a reservoir for the hydraulic fluid (approximately 300 ml per shock absorber). The fluid valves in the piston and in the stationary bottom valve control fluid flow between both cylinders and regulate the damping force. The valves in the piston have the highest proportion of shock absorber damping performance. The material combinations of the main sliding parts in a shock absorber, which are mainly influencing the friction force, are the following:

- NBR-polymer (oil seal) - Cr^{6+}-plating (steel piston rod)
- PTFE/Pb covered bronze (bushing) - Cr^{6+}-plating (steel piston rod)
- PTFE (piston ring) – steel (inner surface of working cylinder)

Thus, the damping force itself consists of two influencing forces, the hydraulic force (oil flow) and the friction force of sliding parts. Since the shock absorber fluid affects the damping characteristics by its viscometric properties and the lubricating properties of various sliding parts, it has a huge influence on vehicles driving, comfort and stability characteristics.

2 DEVELOPMENT AIM AND BACKGROUND

Aim of the present study was to investigate influences of a PAG-based hydraulic oil on the single tribological interacting parts of the shock absorber in comparison to fluids with mineral base oil. Therefore, four different types of shock absorber oils with specific adjusted tribological properties were selected. The lubricant is one of the major influencing factor of the shock absorber performance. For this reason several bench test at KYB Basic Technology R&D Center were conducted to evaluate the interaction of the main sliding parts and oil, mentioned before. Bench test of assembled shock absorbers (ASSY) by use of a servo-hydraulic test machine should show differences in damping force curves. Finally, the oils selected were tested on a public road circuit in a 3 liter rear-wheel drive car. Main target of the test series was to analyze the influence of the oils on driving performance (car stability) and comfort during driving. Currently the influence of shock absorber oil properties, friction between the main sliding parts and the resulting car performance is hardly understood and underrated.

3 TEST OILS AND -PROCEDURE

Four fluids were selected for the tests. Oils No. 23, No. 26 and No. 33 consists of a Group III mineral base oil, whereas oil PAG 1-4 uses a polyalkylene glycol as base stock. Additionally, the

Table 2 Key properties of shock absorber oils tested.

			No. 23	No. 26	No. 33	PAG 1-4
Base Oil		-	Group III	Group III	Group III	Group V PAG
Kinematic	40℃	cSt	12.66	12.82	12.76	12.83
Viscosity	100℃	cSt	4.43	4.502	4.478	3.637
Viscosity Index (VI)		-	317	321	320	184
Flash Point		℃	140	140	148	208
Anillin Point		℃	92.4	91.9	91.6	n.m.
Total Acid Number (TAN)		-	2.06	1.74	1.42	1.46

base oil of PAG 1-4 can be classified as environmentally acceptable lubricant according Ref.[1),4)] and[7)] which means it is readily biodegradable (OECD 301B/F).

The viscometric properties such as kinematic viscosity, viscosity index and flash point are listed in Table 2. Comparing the oils it becomes clearly visible that the kinematic viscosity is nearly in the same range of maximum 12.83 cSt at 40°C. The shock absorber oils based on Group III mineral oil all have a viscosity index above 300 whereas PAG 1-4 shows with 184 a much lower value. Reason for this is that PAG itself has already a comparable higher VI than mineral base oils. Thus, viscosity index improving additives are not needed for many application where PAGs are used. Furthermore, due to the chemical structure of PAGs the flash point is much higher at around 200°C. All lubricants contain additive packages including friction modifiers, antifoaming agents, antioxidants, metal deactivators and antiwear additives (for PAG 1-4 approximately 1.8 wt% in total).

The oils can also classified by the frictional impact. No. 33 and PAG 1-4 have very low friction properties, which will be shown more in detail in the following context. In comparison to these oil No. 26 has the characteristic of increased friction whereas No. 23 oil has the highest friction properties of all oils tested.

The test procedure for the development oils can be consists of three testing steps:

(1) Laboratory tests (pin-on-disc and ball-onthree-plates test principles)
(2) Bench tests (damping force curves by Assy)
(3) Vehicle tests at KYB Suspensions Europe S.A. (KYBSE)

Laboratory bench tests were conducted by means of a self-constructed linear oscillating pin-on-disc tribometer at 30°C oil temperature. Thereby, the pin consists of the same rubber material which is conventionally used for the shock absorber oil seal. The disc counterpart was made from a hard-chrome plated (Cr^{6+}) SUJ2 disc representing the piston rod. The tests run for approximately 40 minutes whereby the sliding speed was reduced in eight steps from 30 mm/s to 0.06 mm/s. This tests support a fast evaluation of the shock absorber oils as well as the oil seal materials and their frictional properties. Additionally, Stribeck-curves were measured with the tribology cell (T-PTD 200) of Anton Paar rheometer MCR 302 at 20°C and −20°C (ball-on-three-plates). Using the material combination PTFE (plate) and steel (ball) the tests can display the characteristics of the shock absorber piston sliding against the inner surface of the working cylinder. In these tests the sliding speed was raised from 4.7E-5 up to 1.4 m/s sliding speed. Damping force characteristics of the test oils were determined by use of mass production shock absorbers with identical setting (valve design) in a servo-hydraulic bench testing machine (Kayaba System Machinery Co., Ltd.). These test were performed with a preconditioned shock absorber at a temperature of 20°C ± 3°C and a stroke of $\Delta x = \pm 25$ mm. A force sensor directly above the piston rod, placed on the mounting point of the machine in *z*-direction, detects changes in the damping characteristics caused by different oils and increased sliding speed over testing time.

In order to evaluate the oil influence on driving performance and comfort, the four oils were tested under authentic road conditions on a public circuit in Spain. Therefore, a 3 liter rear-wheel drive car was selected as test vehicle. KYB Suspensions Europe S.A. currently supplies the front and rear shock absorber for this car. The shock absorber were disassembled and the standard oil was replaced by the oils described before for the tests. During the tests several subjective parameters, such as noise level, harshness, car-, steering- and impact feeling were evaluated by the tests drivers. Additionally multiple acceleration sensors and a microphone inside the vehicle cabin measured data to compare subjective feelings with analyzed, objective test results. The sensor position in the car are illustrated in Fig. 3.

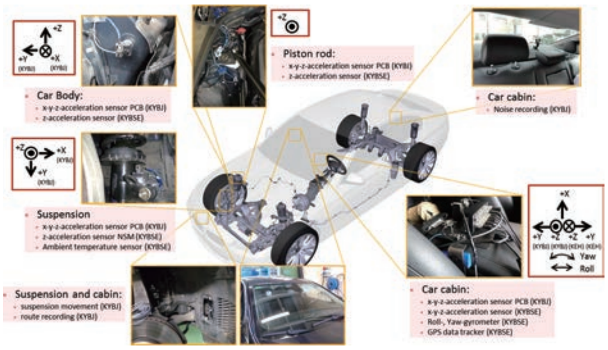

Fig. 3　Sensor positions inside the test vehicle for shock absorber oil evaluation tests at KYB Suspensions Europe S.A.

4　TEST RESULTS AND VEHCILE EVALUATION

(1)Laboratory tests

The test results obtained in KYB pin-on-disc tribometer (Fig. 4) showed a significant difference in coefficient of friction between the four oils.

Oil No. 23 (pink graph) showed, as might be expected due to the additive package, the highest friction in comparison to the other oils. Especially in the low speed area of 0.06 mm/s the friction value μ of No. 23 is approximately four times higher than of No. 33 (blue) and PAG 1-4 (yellow). PAG 1-4 itself has the lowest friction up to 30 mm/s sliding speed. The μ-values of PAG 1-4 range between 0.01 (at 0.06 mm/s) and 0.11 (at 30 mm/s), in case of oil No. 33 up to 0.19. Beside the friction modifier, which is included in the additive package, the higher polarity and therefore better lubricating film formation properties of PAG contributes a reduction in friction. Furthermore, the low friction of PAG 1-4 and No. 33 contributes a reduction in sliding noises. If oil seal material and lubricant are not compatible a so called stick-slip phenomenon can arise. Stick-slip can be described in that case as alternating between sticking of oil seal and piston rod to each other and sliding over each other, with big variations in friction force. This includes in particular the occurrence of noises in low speed area of shock absorber movement and needs to be prevented at any time.

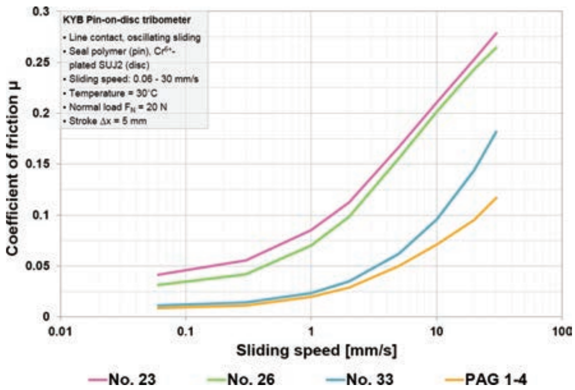

Fig. 4　Coefficient of friction over sliding speed of shock absorber oils tested with KYBs pin-on-disc tribometer.

In addition to the pin-on-disc tests, a second tribometer based on linear sliding principle was chosen to investigate the friction of PTFE-piston and steel surface of the inner shock absorber working cylinder. Furthermore, the influence of reduced temperature on oil friction was evaluate by Stribeck curve recording up to 1.4 m/s sliding speed (see Fig. 5).

Considering the Stribeck curves the different friction characteristics at 20°C are not clearly visible as in the pin-on-disc tests, but rather the high friction oil No. 23 showed comparable low friction when movement starts. All oils were at nearly same friction level and did not show any significant difference in Stribeck-curve progression. The interaction of piston ring and lubricant is of same high interest as the interaction of seal and piston rod as mentioned before. Considering the test results obtained in the rheometer it became visible that the coefficient of friction at the starting point of movement is comparatively high. Especially fluid No. 26 showed

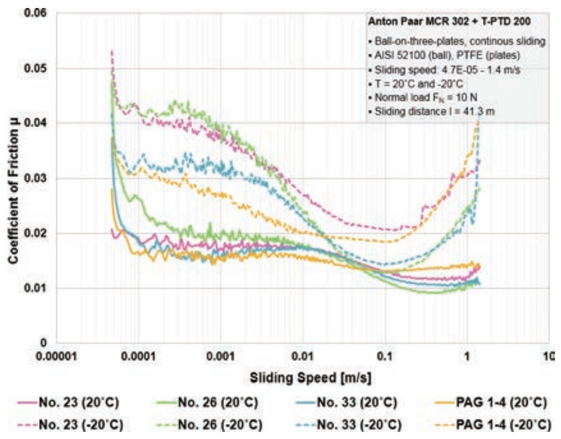

Fig. 5　Stribeck curves of shock absorber oils at 20°C and －20°C.

this behavior. However, PAG 1-4 offered lowest friction up to sliding speed of 0.05 m/s. The area of EHL regime (elastohydrodynamic lubrication) of Stribeck-curve could not be mapped because of a sliding speed limitation of the rheometer used (1.4 m/s). Significant differences in the frictional behavior became visible by an oil temperature reduction to －20°C. Hereby, low the friction oils PAG 1-4 and No. 33 had by far the lowest friction. On the other hand the Stribeck curve progression of PAG 1-4 has a small difference to the curves of mineral base oil samples. Due to molecular interactions PAG changes its cold temperature viscosity faster than mineral oils. This leads to a faster lubricating film formation with increasing sliding speed. Thus, the transition in lubricating regimes between solid, boundary and hydrodynamic lubrication occur faster. Result of this behavior is an obviously higher friction over 1.0 m/s sliding speed.

(2) Bench tests (ASSY)

Furthermore, low friction properties define also the damping characteristics of shock absorber in tests servohydraulic controlled bench tests (Fig. 6). A sensor in the test machine measures the force which is necessary to compress the shock absorber as well as to rebound. Both force values together result in the total damping force depending on damper speed.

Especially the oil behavior in low speed area, from 0.01 to 0.1 m/s sliding speed, is strongly depending on low or high friction properties of the oils. Oils with low friction in laboratory tests revealed also lower damping forces. Here again,

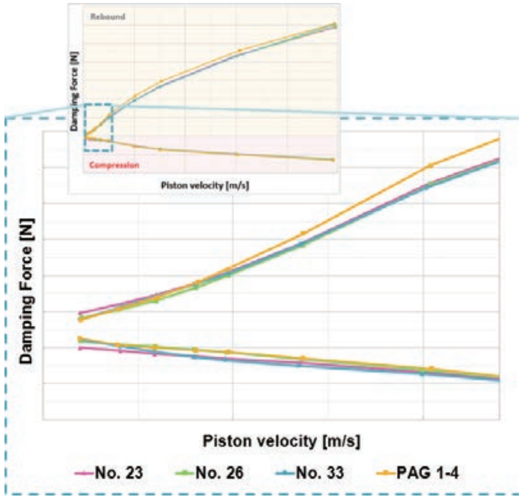

Fig. 6　Damping force test results of rebound and com pression side (ASSY).

low friction has a strong influence on the comfort, perceived road contact and feeling of vibrations. By increasing the piston speed, the damping force increases progressively and the influence of viscometric oil properties raises.

(3) Vehicle tests at KYBSE

Target of the vehicles tests performed in Spain was on the one hand to evaluate the stability and comfort characteristics of the shock absorber oils and on the other hand to compare this subjective data with measurement data recorded by various sensor in the test car. In order to analyze comfort and stability representative test tracks on the circuit were selected. These test tracks showed some clear differences in the cabin noise level recorded by a microphone and also in the cabin and shock absorber acceleration in z-, y- and rolling direction during driving. In total 11 test tracks were selected to evaluate several subjective test parameters and to compare the oils respectively. Fig. 7 shows the results of one selected test track with high level of vehicle dynamics and therefore predestinated for stability analysis. The sensor raw data was prepared by use of LabView and a PSD filter (power spectral density). A PSD-spectrum describes the power variation of a signal over frequency in an infinitesimal frequency band. Considering the diagrams in Fig. 7 and Fig. 8 a lower PSD value indicates higher stability or lower acceleration respectively. In the frequency area of 0.4 to 4 Hz, which is mainly related to greater movements of the vehicle, the high friction oil No. 23 and PAG 1-4 revealed highest cabin stability (Fig. 7 top) and less rolling characteristics (Fig. 7 bottom). This coincides with the subjective evaluation of the track. No. 26 and No. 33 oil were less stable.

The diagrams in Fig. 8 are recorded at a test tracks with very rough and noise-/vibration producing road conditions. Here, the measurements allow conclusions to be drawn about the road contact of the tire. The data reveals less accelerations of the shock absorber and thus of the cabin. Due to the higher stability and low friction characteristics of No. 33 and PAG 1-4 the road contact and therefore steering response could be improved in comparison to No. 23 and No. 26.

Beside stability and driving performance comfort characteristics for driver and passenger are essential. Similar to the car stability evaluation representative test tracks were selected to measure the cabin noise level under different conditions. Hereby, the influence of a sharp road impact (bridge expansion joint) on a highway at 120 km/h (Fig. 9) and a test track with rough road surface at 70 km/h driving speed (Fig. 10) were chosen.

As the test vehicle passed the sharp, metallic bridge expansion joint a clear increase in the cabin noise level could be recognized (see Fig. 9). However, there was a big difference between the four test oils. No. 23 and No. 26 oil were nearly in the same noise range, whereas the oils PAG 1-4 and especially No. 33 could absorb the impact better. This is reflected in a reduction of the cabin noise pressure level by 1.5 dB(A) for PAG 1-4 and 2 dB(A) for No. 33 respectively.

Under continuous driving on a road surface which occurs a high noise level inside the cabin No. 33 and PAG 1-4 oil showed their benefits once again (see Fig. 10). Whereas the noise pressure level over time could be reduced by 2.5 dB(A) with PAG 1-4 in comparison to No. 23 oil, No. 33 oil were nearly an the same level. This effect became perceptible in the frequency area of 2000 – 8000 Hz, which is still audible for the human ear.

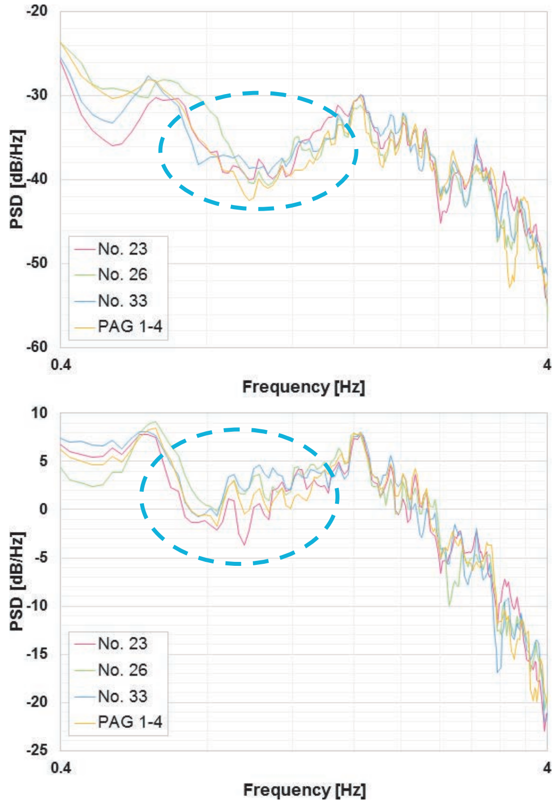

Fig. 7 Stability characteristics: cabin acceleration in lateral direction (y-direction, top) and rolling (bottom).

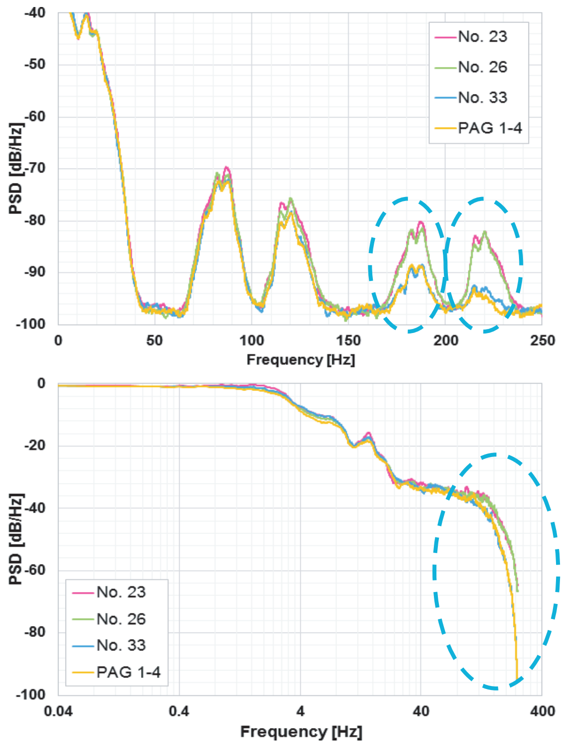

Fig. 8 Vertical acceleration in z-direction of the car: cabin (top) and shock absorber (bottom).

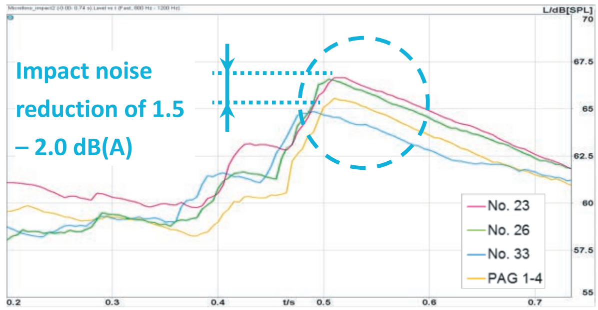

Fig. 9　Bridge expansion joint impact noise (cabin noise pressure level) at 120 km/h.

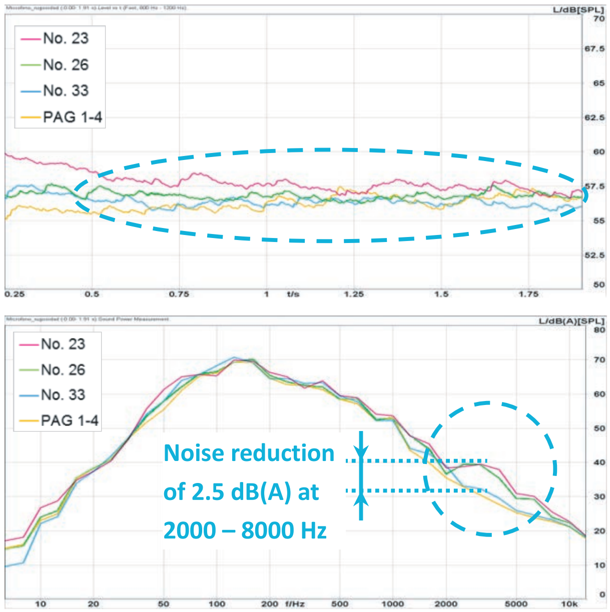

Fig. 10　Background noise inside the cabin: Noise over time (top) and sound power over frequency (bottom) at 70 km/h driving speed.

5　SUMMARY AND CONCLUSIONS

Aim of the study was to evaluate the tribological performance of a polyalkylene glycol-based oil (PAG) as new type of shock absorber fluid. Beside of several chemical and viscometric advantages PAG can be classified as environmentally acceptable lubricant, which takes this oil to a special position on the sales market in comparison to mineral base oils. The laboratory and bench conducted draw the conclusion that PAG 1-4 as fully formulated oil offers very low friction characteristics in combination with the main sliding parts of a shock absorber. Furthermore, the vehicle tests under real driving conditions at KYBSE (Spain) showed a highly promising performance with regard to vehicle stability and comfort.

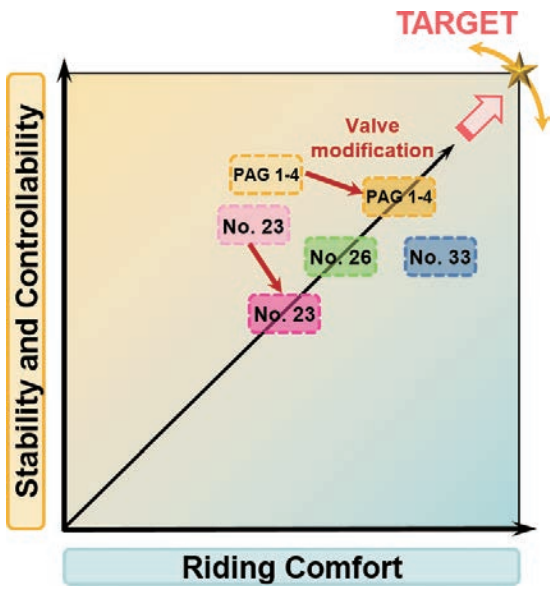

Fig. 11　KYB oil classification regarding stability/controllability and riding comfort.

Fig. 11 shows a summarizing comparison of all oils tested, No. 23 as high friction oil, No. 26 as medium friction oil, No. 33 as low friction oil and PAG 1-4 as low friction oil with an alternative base oil. It is clearly visible that the change of mineral oil based shock absorber oil to PAG 1-4 can have a positive effect especially on the vehicle stability and controllability. In order to improve the comfort characteristics of this kind of oil a modification of the shock absorber piston valve setting would be necessary. However, a very good compromise between riding comfort and stability could be found. Whereas the other oils could only be classified as oil with higher comfort or higher stability, but without improvement in both directions, PAG 1-4 improves both characteristics.

The range of applications for PAG-based fluids is manifold. For hydraulic systems with high stability demands or special requirements on the base oil (flashpoint, hydrolytic stability) and even for shock absorber systems in the motorsports sector PAG can offer a promising alternative to commonly used oils. Furthermore, the use of an environmentally acceptable oil based on PAG could in future be a useful sales argument with regard to the global environmental awareness in many industrial areas.

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2007年入社．技術本部基盤技術研究所材料研究室．ショックアブソーバ用材料の研究開発に従事．