THE QUALITY ASSESMENT AND TOXICITY OF SELECTED SERVICE FLUIDS BASED ON GLYCOLS

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Abstract: *Eight samples of selected service fluids were evaluated from the view of their quality. From results is perceptible that physical-chemical parameters which are declared by manufactures are fulfilled (correspond with DOT), in case of higher water content and extreme conditions (especially minus temperatures), the function of brakes must not be ensured although is vehicle equipped with ABS,ESP etc. From the research of toxicity of selected samples is perceptible basic glycol chemicals are non-toxic or, maximum low-rate toxic, but the acute toxicity (LD 50) of selected brake fluids (especially from DOT 3 classification) is relatively high and largely is it medium toxic substances. The using of unsuitable brake fluid could generate heavy impacts not only from the view of the road safety but the influence of service mechanic´s and driver´s health who are in touch with service fluids during checking or oil changing.*

Keywords: GLYCOLS, BRAKE FLUID, BOILING POINT, VISCOSITY, SAE, DOT, TOXICITY, LD_{50}, LC_{50}

1. Introduction

According to the historical sources, just American company Duesemberg, who presented racing car with hydraulic brake in 1920 on the motor-show in New York. One year later company racer Jimmy Murphy won with this car Trophy of France in Le Mans and experts mean that hydraulic brakes decided about winner. Since the time, development and research of these fluids have been waged.

First fluids were made from materials based on the ricin oil and alcohols. In fifties of twentieth century, these fluids were gradually replaced by materials like glycoethers and polyglycols. In sixties (1966), company Citroen like first producer of cars started use mineral brake fluids and on the start of eighties is followed by Rolls-Royce. Brake fluids based on the mineral oil weren´t used by others producers of cars. The main problem was that is possible to destroy elastomers by casual mix of two different types of fluids. Brake fluids based on the silicon are used in special cars of US army. During sixties and seventies of twentieth century brake fluids with higher power were revealed, they are based on the polyglycolester and ester of boric acid or silicous acid.

2. Composition of brake fluids

Basic components of modern brake fluids fulfilling at least DOT 3 are short ethers in a form of di- and tri- (alternatively tetra-) ethylenglycol (see Fig. 1).

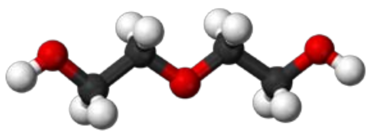

Figure 1. Structural formula of diethylenglycol ($C_4H_{10}O_3$) [18]

These have one end of oligomer molecules end by ether bond with methyl, ethyl or buthyl end. These molecules provide the high flash point (about 250°C), low volatile characters (low tension of vapours) and the relatively good viscosity.

Modern brake systems have requested, except the high flash point of brake fluids, especially the lower hydroscopicity, lower dependence on the temperature especially temperature below zero which is important for vehicles equipped with the ABS, ASR or ESP. It relates with the relatively higher increase of material price which fulfil these characters.

With the regard of variations possibilities of di, tri and tetra ethylenglycol, is definite brake fluids of the most famous world producers (BASF, DOW, CLARIANT etc.) do not have same composition, because of different materials availability for these concerns and the patent restriction. Even particular samples of one brake fluid type of some producer can have different variations according to the availability of and especially particular substances price during year, which are used for brake fluid during year.

Owing to price result, composition of brake fluids is often "set" by relatively easy and well available molecules of Diethylenglycol a Butlyglycolu, their higher dosage is limited by harmful effects for health (symbol Xn) and lower boiling point (<250°C). These negative aspects does not relate with Polyethylenglycols, which significantly increase viscosity of brake fluids. Current brake fluids contain basic fluid Triethylenglykol monomethylether orthoborate (current brake fluids content approximately 30 % vol and more). In tenth of unit of weight percent, brake fluids content inhibitors of corrosion on ussuall metal construction materials of brake system, in the main substances based on organic bounded nitrogen , very often in synergy combination with Triazols.

3. Physical-chemical properties of brake fluids

For the description of of brake fluids physical-chemical properties based on glycols exist several international , national and company norms and specification for example SAE J 1703, SAE J 1704, FMVSS CRF 571.116 DOT 3, DOT 4 a DOT 5.1, ISO 4925 Class 3, 4, 5.1, 6, VW TL 766/ -, X, Z a VW 50 114 [1], [20]. In these norms are presented basic physical-chemical parameters and strict procedure of their evaluation. It is assessment of colour, boiling point (dry and wet boiling point) oxidation and chemical stability, kinematic viscosity, evaporation abilities, toleration with elastomer, pH factor, miscibility, anti-corrosive effects, procedure of imitation of service performance. The limit values of these parameters for DOT 3 and DOT 4 fluids are in table 1.

4. Materials and metdods

The assessment of brake fluids is often proved in laboratories of brake fluid producers. Results of mutual comparation physical-chemical parameters of brake fluids from particular producers are fragmentary. Therefore, for the assessment of brake fluids quality attributes, 8 brake fluids samples, class DOT4 from different producers were bought. The water content was ascertained by titration methours and boiling point by distillation curve. The results are stated in graph (fig. 2).

The figure shows that the experimental measured values the brake fluid samples DOT 4 comply with dry and wet boiling point according to the standards (SAE, ISO, VW). But in the SAE standard, in relation to thermal-viscosity properties, the limit values of kinematic viscosity are specified for two temperature conditions: for at –40 °C (acc. at SAE max. 900 mm^2/s) and at +100 °C (acc. at SAE min. 1,5 mm^2/s). The kinematic viscosity values for these two temperature limits apply only to new brake fluids with water content up to 0.2 %. The effect of service hygroscopicity on viscosity properties of glycol brake fluids has not been specified.

Table 1. Parameters according to the DOT4 [20]

Physical-chemical properties of brake fluid class DOT 4		
Visual aspects	clear, without mechanical pollutions	
Boiling point [°C]	- dry - wet	min. 230 min. 155
Kinematic viscosity [mm^2/s]	- at - 40°C - at +100°C	max. 1800 min. 1,5
Corrosive test (weight decrease on samples) [mg/cm^2]:	- AmE alloy - tin-plate metal - steel, cast iron - copper , zinc - brass	max. 0,1 max. 0,2 max. 0,2 max. 0,4 max. 0,4
Rubber cuff (SBR rubber): - decomposition - hardness decrease(IHDR) - increase of cuff diameter [mm]		none max. 15 max. 1,5
Effect on rubber: a) 70°C (natural rubber): - hardness decrease [IRHD] b) 120°C (rubber SBR)*: - hardness decrease [IRHD]		max. 10 0 - 15
Low temperature liquidity: a) -40 °C (144 hour. test): - start of bubbles expansion [s] - time of bubble expansion [s] b) -50 °C (6 hour. test): - start of bubbles expansion [s] - time of bubble expansion [s]		max. 5 max. 10 max. 5 max. 35
Toleration with water(3,5 % vol. H_2O): a) -40 °C (22 hour test): - visual aspects b) +60 °C (22 hour test): - stratification - sediments [% vol.]		comply without stratification max. 0,15
Mutual miscibility		7,0 - 11,5
Imitation of service performance: - at 70 °C a 120 °C, - pressure 6,865 MPa, - frequency : 1000 travels/hour		- this test is not performed in Czech republic

So that four samples of DOT 4 class brake fluid with water content of 0.2 % by weight, 1.7 % by weight, 3.6 % by weight and 5.9 % by weight were prepared in the lab. The water content in brake fluid samples was measured by the Karl Fischer methours according to the company standard [4] in Velvana inc., Velvary.

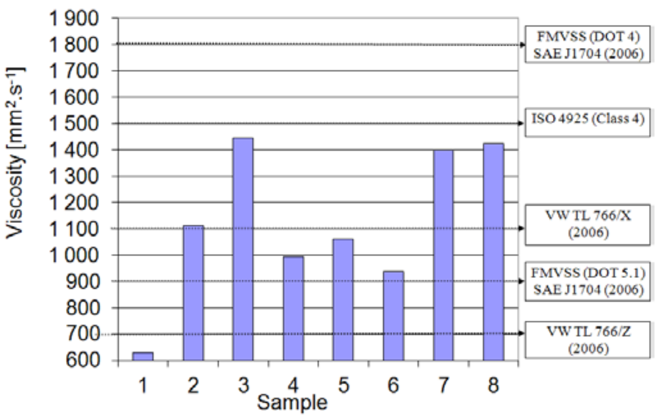

Figure 2. Boiling point of brake fluids class DOT4

Ubbelohde viscosimeter was used for the determination of the brake fluid kinematic viscosity.

The kinematic viscosity measurement of the aforementioned four SYNTOL HD 265 PLUS brake fluid samples was performed at -40 °C and +100 °C temperatures. Temperature treatment of the viscosimeter by means of the LAUDA type RL6 cryostat made possible the viscosity measurement at -40 °C.

The kinematic viscosity has been calculated from the following formula:

$$\nu = kt \qquad (1)$$

where: k – constant of calibration (viscosimeter for 100°C: k = $0{,}01000.10^{-6}$ m^2/s^2, viscosimeters for -40°C: $k = 3.001 / 3.005 / 3.006 \times 10^{-6}$ m^2/s^2),

t – time.

5. *Results and discussion*

Summary results of the SYNTOL HD 265 PLUS brake fluid kinematic viscosity measured at –40 °C are plotted in Fig. 4 and at +100 °C temperatures are plotted in Fig. 3.

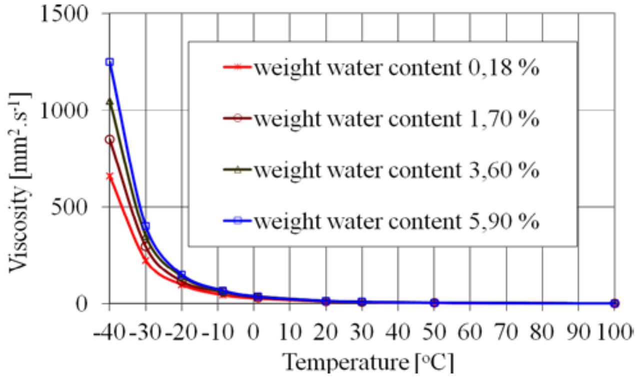

Figure 3. Brake fluid viscosity

The figures 3 shows that the brake fluid viscosities vary not only with temperature (this fact has been known), but also with the change of water content (this fact has not been known up to now).

The regrese curve running through the values measured at –40 °C temperature (Fig. 4) shows that even low increase in water content (by about 5,9 % by weight) raised the value of the brake fluid viscosity from 645 mm^2/s up to 1240 mm^2/s, i.e. by 97 %. Such rise in viscosity is undesirable for the service because of hypothetical consideration that the rise can result in adverse multiple longer reaction time of the brake system. This fact can have far-reaching consequences as far as the operational safety of the vehicle is concerned especially in winter season, immediately after starting the vehicle. This regime is extreme; therefore the results measured should be regarded as extreme ones as well.

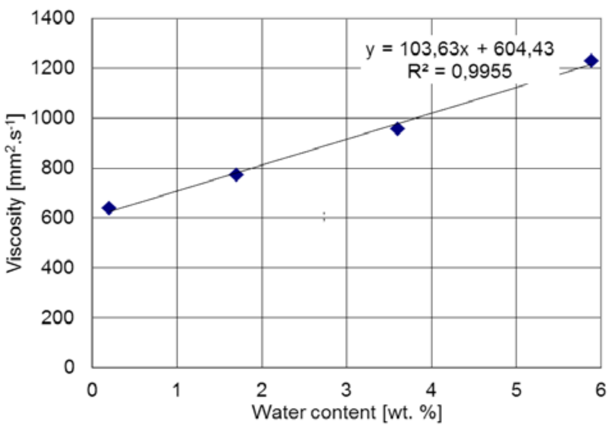

Figure 4. Brake fluid viscosity - water content relation for –40°C

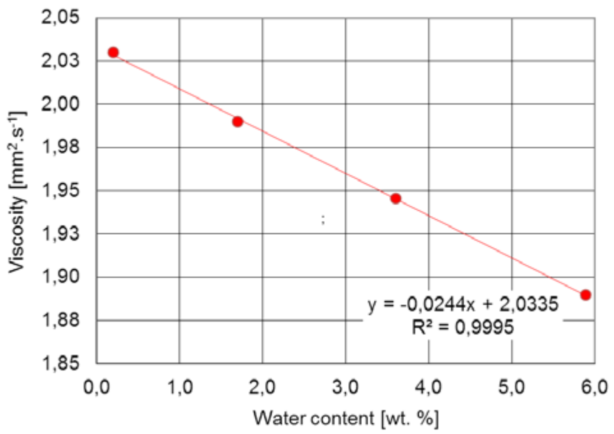

Figure 5. Brake fluid viscosity - water content relation for +100 °C

The regrese curve running through the values measured at +100 °C temperature (Fig. 5) shows that with the rise in water content the brake fluid viscosity decreases. The decrease in water content (by 5,9 % by weight approximately) reduced the brake fluid viscosity from 2,03 mm^2/s to 1,89 mm^2/s. This decrease in viscosity is also undesirable for the operation because of adverse effect on the transmission of effort from the brake pedal to control members, deterioration of lubrication function, etc. The braking regime, when brake fluid temperature of +100 °C is attained, can be rather normal in service. Far more extreme regime of vehicle braking, when the brake fluid temperature reaches 155 °C [1], can occur in practice (when driving down gradual decline, during abrupt intensive braking in high initial speed, etc.).

From this is perceptible, using of insufficient fluid may have impacts on the brake system function, it means incalculable consequences from the view of service safety

6. The toxicity of service fluids

Instead of chemical properties of vehicle service fluids as well as their toxicology properties are important. Toxicity is property of chemical compounds which cause poisoning of animals or people who absorb, ingest or inhale these substances. Toxicity is checked by special tests. Nowadays exist a high number of toxicology tests, which is possible divide into [3], [5], [19]:

- time of exposition (acute, semi- acute, chronic),
- generation of the test system (standard tests, micro-biotests and biosensors, bio-probes andbiomarkers),
- trophic level of the test organisms (producers consumers and destruenty),
- the test matrix (water, soil, air, sediment, waste, chemical, etc.)
- range of test organisms (single-and multi-species natural populations and laboratorycultures mix),
- type of test sample (pure chemical (hydrophilic, hydrophobic, volatile), a mixture of substances known and unknown, largely unknown natural samples and mixed, etc.),
- the methours of sample preparation (defined concentrations of chemicals, testingextracts of natural samples by extraction with organic solvents, water, different pH,temperature, etc.),
- degree of complexity of the detection system (from easiest to most difficult - enzymes,biosonds, cell and tissue cultures tives organism, population, and mezocosmosmicrocosm, field xperiments),
- the way in scoring (lethal effects (mortality, immobilization), sublethal effects (behavior of organisms - such as speed and direction of movement), evaluation ofphysiological activity (photosynthetic assimilation, enzyme activity, effects on membranes, assessment increases - root length, number of cells in the population, the weight of organism susceptibility to attack diseases, pests or parasites, etc.),
- reproductive activity, malformations and teratogenicity, etc.),
- special tests to evaluate the risks in the environment (for cases where a particularinterpretation is necessary to provide non-standard effects on the test organism, egtrophy, mutagenicity and genotoxicity of bacteria, genotoxicity on plants, wildlife andfish, teratogenicity, for example amphibians (Xenopus laevis), embryotoxicity and reproductive tests on fish, crustaceans, amphibians, birds and other).

For the distribution of materials according to toxicity, several scales are used and according to which the organism is causing substance.

Most substances are dividedaccording to exposure to humans and bees. In the case of toxicity in humans is the container used substances given these tags T+ (the substance is highly toxic), T (toxicant), Xn (substance harmful to health), Xi (irritant), C (CORROSIVE) andunlabelled (substances where toxicity is indicated).

In order to measure and compare the toxicity were introduced special abbreviatedscale LD (Lethal dose), LC (Lethal concentration), LT (tempus letale medium), ET (tempus effectivum medium) and similar.
In describing the deadly dose of chemicals compounds are used designations [3], [5], [17], [19]:

LDXy (z) [mg / kg] (2)

- X - denotes the percentage of test animals died after receiving a dose died as a result,
- Y - denotes species on which the test was performed,
- z - of-refers to the methours of dosing (inh.) - Inhalation (inhalated), (inj.) - injection (oral)orally.

For example LD_{50} (oral, rat): > 18,056 mg/kg dose indicates filed orally, have died atall 50 % of test animals (rats). Classification of toxic substances under LD_{50} values are listed in Table 2.

Tab 2. Classification of toxic substances under LD_{50} values

Chemical	LD_{50}
Super toxic	5 mg/kg and less
Extremely toxic	5 - 50 mg/kg
Highly toxic	50 - 500 mg/kg
Moderately toxic	0,5 - 5 g/kg
Few toxic	5 - 15 g/kg
Non-toxic	15 g/kg or more

In describing the deadly concentration of chemical compounds in inhaled air is used foridentification [3], [5], [17], [19]:

LCXy [mg/l] or [ppm] (3)

where:

- X - denotes the percentage of test animals as a result of the inhalation concentration died,
- Y - denotes animal species on which test was performed.

For example ppm/4h LC_{50} = 64000 (rat by inhalation) denotes the concentration and time of inhalation, at / have died after 50 % of monitored animals (rats).

Classification scale of deadly concentration of chemical compounds in the air inhalation LC_{50} is listed in Table 3.

Tab 3. Classification scale LC_{50} for gaseous substances [x] [5], [19]

Chemical substance	LC_{50}
extremely toxic	10 ppm
very toxic	10 -100 ppm
toxic	100 - 1000 ppm
Low toxic	1000 - 10000 ppm
practically non-toxic	1 - 10 %
practically harmless substance	10 %

[x] - by Marhold for gaseous substances, rat, 4 h exposure time

Toxicological information about each product, the manufacturer must indicate in the safety data sheet. In this paper also presents data about the ecotoxicity under the designation LC_{50} (concentration of an aqueous extract of the waste or waste solution, soluble in water, at which 50 % died of test organisms [mg./l]), IC_{50} (the concentration of an aqueous extract of the waste or waste solution of soluble in water, which causes 50 % inhibition of cell density [mg/l)), EC_{50} (concentration of an aqueous extract of the waste or waste solution, soluble in water, which causes 50 % of the Daphnia immobilization [mg/l]) and more.

Toxic effect of substances on the test organism by concentration values are specified LOEC (Lowest Observed Efect Concentration), which represents the lowestconcentration of test sample in which the effects are observed, and the NOEC (no-Efect Observed Concentration), which represents the highest concentration of test sample which do not result in any observable effects.

To test the toxicity of substances is used a wide range of organisms (bacteria, cyanobacteria, algae, higher plants, water and soil organisms, mosses and lichens, butalso animals). The experimental toxicology of “classical” laboratory animals are most commonly used mouse (54 %), rats (39 %), guinea pigs (2 %), rabbits (2 %) and others (dogs, cats, frogs, birds, fish, primates and similar.) about 3 %. The substance be given orally, by injection, dermally, or inhalation. Work with laboratory animals must beguided by ethical principles for working with laboratory animals and each experimentusually must be approved by the Ethical Committee of the workplace.

Among the best known and most widely used tests in laboratory practice, we include standard laboratory tests of acute and chronic toxicity.

In acute oral toxicity test for the detection of harmful effects of the substance (LD_{50}) after short exposure, or after several doses during the 24 h with observation timeminimum of 14 days [mg/kg]. Carrier of toxic substances is water ,olive oil, etc. Changes are monitored in behavior, appearance, weight, etc.

Acute dermal toxicity test is to determine the harmful effects (LD_{50}) after short-termdermal application [mg / kg]. Graded doses (concentration) is applied to the back after depilation (area about 10% of body surface). Test of acute inhalation toxicity of gases, vapors and aerosols (LC_{50}) is to determinethe effects of harmful substances in the breathing air after one attempt uninterruptedexposure [g/l]. In this test the animal is immobilized, graded doses (min. 3 concentrations with exposure time of ~ 4 h inhalation chamber.

Acute dermal irritation test is the detection of reversible inflammatory changes in theskin (irreversible burns) when applied to the back depilation (area approximately 6 cm^2). Apply rule 0.5 ml of liquid or 0.5 g solids at minimum three animals. Exposure timeis 4 hours, followed by washing and observing changes after 30 to 60 minutes, then every 24 hours up to 14 days.

Acute eye irritation test is the detection of harmful effects after application of the substance on the outer surface of the eye. It evaluates the reversible changes(irritation) and irreversible changes (burns). The aim is to find information of the performance of the substance on the eyes and adjacent mucosa. Test is minimum for 3 animals (usually albino rabbits). The test is performed with dose of 0.1 ml (liquids), 0.1g (solids). The conjunctival sac of one eye, second eye is control. During the test, are used local anesthetic. After 24 hours, the eye is washed with water and then are observed (under the magnifying glass, microscope) changes after an hour, then after 24, 48, 72 hours.

Determination of chronic toxicity (repeated doses of low concentration) is important in terms of setting thresholds for long-term contact with foreign substances. Sense of these tests is the observation of laboratory animals for the major part of their lives. The results are used to determine significant effects on the genetic material of cells (carcinogenicity, mutagenicity, reproductive toxicity, allergic reactions, etc.).

Table 4 shows the values of acute toxicity of selected chemicals used in the operating liquids and Table 5 shows the values for acute toxicity of selected operating brake fluids

Table. 4 Acute toxicity values of selected chemicals used in operating fluids [6],[7],[8],[9],[10],[17], [19]

Substance	Oral LD_{50} [mg/kg]	Dermal LD_{50} [mg/kg]	Ecotoxicity LC_{50} [mg/l]
MEG	> 2.000 (K)	3.150 (K)	18.500 (96 hours, fishes)
MPG	19.400 - 36.000 (P)	20.800 (K)	51 600 (96 hours, fishes)
DEG	12.565 (k)	11.890 (K)	32.000 (96 hours, fishes)
TEG	15.000 (P),	2.000 (P)	10.000 (96 hours, fishes)
TEGBE	2.000 (P)	3.480 (K)	500 (72 hours, fishes)

Where: MEG - Monoethylenglykol, MPG - Monopropylenglykol, DEG - Diethylenglykol, TEG - triethylenglykol, TEGBE - Tetraethylenglykol, P - mouse, k - rat, K – rabbit

From the table 4 is perceptible, glycol chemicals which are used in service fluids are non – toxic or low intension toxic. Only the MEG is from a group of medium- toxic, but this chemical has not used yet in brake fluids. MEG is basic component of cooling fluids, which are used in a high rate today.

Table. 5 Acute toxicity values of selected brake fluids [11],[12],[13],[14],[15],[16],

Substance	Oral LD_{50} [mg/kg]	Dermal LD_{50} [mg/kg]	Ecotoxicity LC_{50} [mg/l]
Syntol HD 205 and HD 265	>2000 (k)	non-irritating	>100, (96 hours, fishes)
LUCAS DOT 4	>5000 (P)	not listed	not listed
AG DOT3 and DOT4	>5000 (K)	>2000 (K)	>100, (96 hours, fishes)
CINOL DOT3 and DOT4	2000 (k)	non-irritating (K)	>100, (96 hours, fishes)
HEKRA DOT3 and DOT4	2000 (P)	not listed	596/100, (96 hours, fishes)
DONAX YB and ZB	2 000/5000 (k)	2 000/5000 (K)	>100, (96 hours, fishes)

The table 5 have shown, that acute toxicity of selected brake fluids (in the main DOT 3, oral way) in compare with their basic fluids, is relatively high and largely chemicals which are medium toxic chemicals. The main reason is that brake fluids contain next chemicals which ensure oxidation stability physical- chemical properties (high boiling point, low dependency of viscosity – temperature, corrosion protection, etc.). The new brake fluids of class DOT4 have lower toxicity (low toxic chemicals). Is necessary to state, in vehicle service fluids are many dangerous chemicals which are legistatively monitored. For example it is ester 1,2-benzendikarboxyl phthalate acid (PAE).

The research which has been performed at University of Defence in Brno, in cooperation with Mendel University in Brno, confirmed the presence of phthalate acid esters. Their danger is based in exposition of phthalates from these serice fluids which is possible by inhalation, oral way, or skin resorption [2].

Phthalates and their metabolites are presented like potential harmful for human and the environment with regard to their hepatoxic, teratogen and cancerogenic character).

The legislation of EU does not occupy by phthlates and other pollutants which are contained in service fluids of vehicles. However it is material which mechanics and drivers are in touch with, during their changing and checking.

7. Conclusion

The present writers are concerned with the selected problems of brake fluids research. From the operational point of view the boiling point and viscosity are the most significant physical-chemical value of glycol brake fluids.

The purchased DOT 3 and DOT 4 class brake fluid samples comply with dry and wet boiling point according to SAE and DOT standards. This observation is certainly good news for drivers. No fundamental differences of the boiling point between domestic and foreign brake fluid products have been found out. In relation to thermal-viscosity properties, the limit values of kinematic viscosity are specified for two temperature conditions: for -40 ^{o}C and for +100 ^{o}C.

The kinematic viscosity values for these two temperature limits apply only to new brake fluids with water content up to 0.2 % by weight approximately. The effect of service hygroscopicity on viscosity properties of glycol brake fluids has not been specified. Four samples of the SYNTOL HD 265 PLUS brake fluid with water content of 0.2 % by weight, 1.7 % by weight, 3.6 % by weight and 5.9 % by weight were prepared in the lab.

The results have been showed that the brake fluid viscosities vary not only with temperature (this fact has been known), but also with the change of water content (this fact has not been known).

From the paper is perceptible, brake fluids in compare with other service fluids haven't strict chemical structure, it relates with difficult development of their requirements in the past century. The close analysis of brake fluid market has shown fulfiling basic reqirements is sufficient. However is necessary to distinguish between close-type fluids which fulfil DOT 4 norm, when a few of them fulfil requirements for low viscosity while negative temperature like tested SYNTOL HD 265 PLUS. Based on his parameters is displayed complicated issues dependence viscosity not only on the temperature but also on the water content. From this is perceptible, using of insufficient fluid may have impacts on the brake system function, it means incalculable consequences from the view of service safety

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