Improving the Precision of Specifications by Evaluating the Influence of Test Parameters on Tribological Results
– A Synthesis from a Series of International – Round Robin Tests–

Mathias Woydt[1] and Hartwig Weber[2]

1 Federal Institute for Materials Research and Testing (BAM), D-12200 Berlin (Germany) [Chairman of DIN 51834-1 to -9, ASTM D5706, D5707 and D6425]
2 Optimol Instruments Prüftechnik GmbH, Friedenstr. 10, D-81671 Munich (Germany)

Abstract: A series of cooperative interlaboratory tests (round robins) was conducted in 1997, 1998, 1999, 2000, 2001, 2002 and 2003 by the DIN 51834 Working Group on Tribological Tests in Translatory Oscillation Apparatus. The statistical analysis of these test results shows the influence of cleaning solvent, machine model and evaluation criteria on the tribological properties of the lubricants tested. Coefficients of friction and wear results are ranked according to the effects of ten different cleaning solvents, where isopropanol gave the lowest values and isoparaffin solvents the highest. The effect of machine model on coefficients of friction varied from about 0.2 % to 0.9 % of the mean. Wear results were not affected. The tests also showed that the seizure criteria and methods of measuring wear required for in the test procedure do not provide a suitable measure of the tribological properties of some lubricants. The precision was improved by introducing a grease apply caliper as well as an increased stroke to 1,5 mm and running-in.
The temperature does not affect the precision of the oil test procedure.

Keywords: tribology tests, ep, wear, friction, lubrication, oil, grease, SRV

Introduction

Industry seeks to use test systems that enable a qualitative or even semi-quantitative correlation between cheap model tests and expensive and time-consuming component or product testing [1]. There is a strong demand today for test procedures that can rapidly screen potential lubricants and materials before system-level life tests are performed.

Tribometers serve to characterize friction and wear behavior of materials and coatings or/and interaction with lubricants, greases, base fluids, additives and gases. They represent the basis for tribology-oriented development of materials and lubricants or for quality assurance. They also serve to check the conformity of a product with a specification.

The principal advantage of using ASTM, ISO, DIN or other standard test methods is that they have been carefully evaluated by experts and their procedures have been carefully documented. Their repeatability not only depends on the design quality of the test equipment, but also on the knowledge about the influence of test parameters and/or operating conditions. In other words: a test procedure can only be as good as the knowledge about the influence of test parameters and/or operating conditions is advanced.

This is the key question, because the trust in and transferability of test results depend also on the repeatability and reproducibility of the model test procedure.
Round Robin tests are necessary to prove regularly (usually every two years) for certified labs, that machine and operator produce credible results. Since 1996, the working group for DIN 51834, part 1 and part 2, which both were issued April 1997, has decided to organize and run one round robin test every year in order to be able to show and quantify in the future systematic and other errors.

This paper summarizes the findings from the RR tests of 1997, 1998, 1999, 2000, 2001, 2002 and 2003 as well as the discussions within this working group, which may be transferable to other tribology test procedures.

Test Procedure

Standardization

The results presented in this paper were elaborated using standardized test equipment under linear-oscillating motion. According to the policy of Deutsche Institut für Normung (DIN), DIN 51834 [2], part 1, issued April 1997, describes only the technical recommendations for the equipment. Since May 1999 DIN 51834, part 1 and part 2 are available from DIN as official translations into English. DIN 51834 [3], part 2, describes the actual test procedure for oils. The precision statements in part 2 are based on a round robin test performed in 1988 with three oils, but were refined in 2000/2001. The first SRV-test method appeared in 1995 as ASTM D5706 and D5707.

The test principle was developed in 1968. After more than 35 years of experience with more than 240 test machines worldwide, a number of test procedures have been developed and standardized (see Table 1). Additionally, these standards are transferred from the ASTM methodology into DIN and vice versa.

Table 1 - *Standard Test Procedures Referring to the High-Frequency, Linear-Oscillating Test Machine*

Type of Lubricant	DIN		ASTM
Oils, fluids	51834, part 2 (white print)	⇒	D6425-02
	51834, part 3 (white print)		
	51834, part 4 (under preparation)		
Greases [4]	51834, as future part 6	⇐	D5706-02
	51834, as future part 7	⇐	D5707-03
Needle and sinker oils	E 62193, part 1 (white print)		
Solid bonded films	E 51834, part 8 (draft 2003)	⇒	Dxxxx
Grease lubed polymers	E 51834, part 9 (Draft 2002 from TRW)		
Test apparatus	51834, part 1 (white print)		

Based on these efforts, more and more OEMs base their technical requirements on tribological tests with SRV [5, 6] and the next step relies on piston ring/ cylinder liners.

Test Equipment

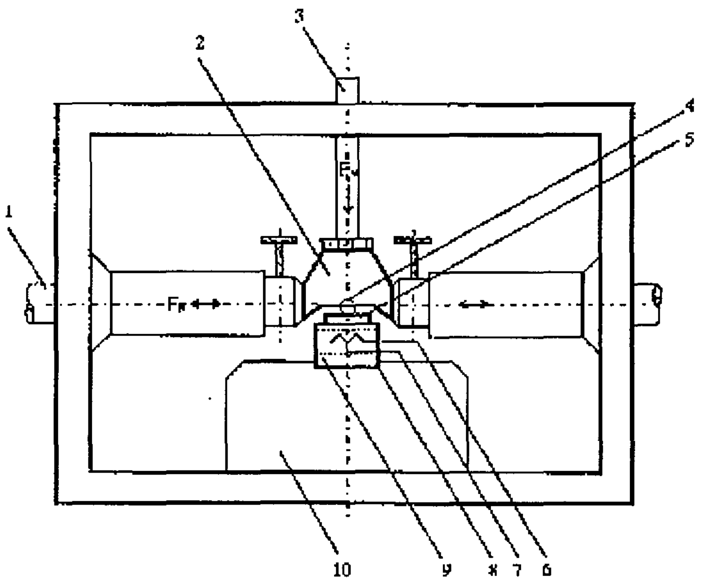
In the basic test configuration, an upper test specimen is rubbed against a lower specimen (see Figure 1). Few milligrams of lubricant (0.3 ml) or grease may be placed into the tribo-contact. After lubricant has been placed on the test specimen and these have been installed in the test chamber, the normal force is applied mechanically to the upper specimen in a direction normal to the direction of motion at a given test frequency and stroke.

The friction force is measured continuously by means of a piezoelectric load cell under the lower specimen holder, which is attached to a rigid test block. The wear scar and track dimensions are determined after a given test duration by optical microscopy or stylus profilometry.

The test block and the holder can be heated to +295°C (optionally up to +900°C) and cooled to control the temperature of the lower specimen.

DIN 51834, part 2, requests a ball as upper specimen and a disk as lower specimen, both made from 100Cr6H (DIN 1.3505), equivalent to AISI 52100 (UNS G52986) with HRC 62 ± 1. AISI 52100 is a vacuum arc remelted (VAR) steel according to DIN EN ISO 683-17 or ASTM A295/E45 with low inclusions and spherodized annealed to obtain globular carbide. The lower specimen is a lapped disc with a diameter of 24 mm and a height of 7.85 mm. The topography of the disk is lapped and determined by four values: 0.5 μm<Rz<0.650 μm; 0.035 μm < C.L.A. (Ra) <0.050 μm, 0.020 μm<Rpk<0.035 μm and 0.050 μm<Rvk<0.075 μm. The upper specimen is a polished ball with a diameter of 10 mm and a roughness of C.L.A. < 0.025 μm.

In addition to part 2, DIN 51834 [7], part 3, describes a procedure using all kinds of materials and coatings. In other words, using DIN 51834, part 3, with AISI 52100 specimen means to test in accordance with DIN 51834, part 2. Other test geometries can be a horizontal cylinder, for Hertzian line contact, or an upright cylinder or ring for area contact.



1 Drive rods
2 Specimen holder (top) (20 different adapters and holders available)
3 Loading rods
4 Specimen (top)
5 Specimen (bottom)
6 Temperature sensor
7 Heating
8 Specimen holder (bottom
9 Piezo measuring element (calibrated according to ISO 9000)
10 Receiving block

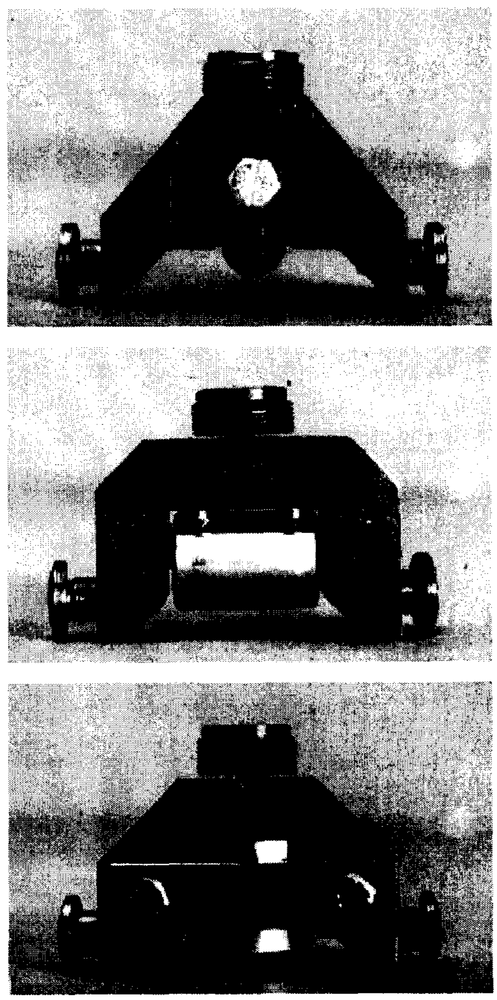


Figure 1: *Schematic view of the SRV® Test Arrangement and the Test Chamber*

The characteristic feature of fretting wear compared with other types of wear lies in the intensive interaction between the surfaces, transfer of debris, mode of vibration and wear.

Influence of Test Parameters

The influence of only those test parameters will be discussed which are assumed to determine the test results, such as cleaning procedure, model of test machine and test pieces. The relative humidity has a friction and wear determining influence for oscillating [*8*] contacts under unlubricated conditions, but was considered to have only a minor influence under lubricated conditions.

Round Robin Tests

The round robin tests performed in 1997 with 19 participants, 1998 with 22 participants as well as 1999, 2000, 2001 and 2002 with 25 to 36 participants each used formulated or only base oils or oils with a low EP capacity in order to show specific properties of the test procedure. The test kits with samples, test oils and a diskette for saving results were sent to the participants in March. The time available for running the round robin tests always was 3 months, from 1st April to 1st June. 80% of the participants returned the results on diskette.

Table 2. *Compilation of the Test Oils Used for Round Robin Tests*

Test oil	Viscosity @40 C [mm²/s]	Viscosity @100 C [mm²/s]	FZG A/8,3/90
CEC RL175/3, (Nov. 1995)	169.3	15.8	
Polyalkyleneglycol 50-100B	139.0	27.0	
Polyalkylenglycol PAG46-2 (VL972, PANA, PAS)	47,4	9,9	
Paraffinic ISO VG 220, Additivated	202.5	17.6	11
BP Energol GR-XP 220	210	18!"	
SHELL Omala HD	220	25.5	>12
MOBIL SHC 500 (VG46)	46.0	8.5	>12
Synthetic Ester (blue angel) Kajo-Bio-HEES-S (VG46)	49.5	9.7	12

The results of the ´97 and ´98 round robin tests have been published in [*9, 10*] and those of 1999 and 2000 in [11] after having been approved by the workgroup in its yearly September meeting. The participants received a certificate with the results of their own lab and the test data of all other participants in anonymous form. Table 2 compiles the basic properties of the oils used.

The polyalkyleneglycol was butanol initiated with an ethylene-/propyleneoxide ratio of 50:50. The German environmental label "blue angel No. 79" Kajo-Bio-HEES-S was attributed to this hydraulic oil because it is biodegradable and contains eco-friendly additives. The "SHC 500" is a PAO-based synthetic anti-wear hydraulic oil. The " SHELL Omala" is a PAO-based (small ester content) synthetic industrial gear oil for CLP-requirement. The BP Energol is a mineral-based CLP industrial gear oil. The polyalkyleneglycols were butanol initiated with an ethylen-/propylenoxide ratio of 50:50 and were tested as unadditivated base oil and additivated. The "CEC RL175-3" is a mineral based reference test oil for Timken- and 4-ball-testers with an EP-package.

Round Robin Test Results

The collection of the data on floppy disk and the import into a database facilitated the statistical analysis and the identification of clusters as a function of parameters. The analysis of the RR97 and RR98 was performed according to DIN ISO 5725 and DIN EN ISO 4259. RR99, RR2000 and RR2001 were additionally evaluated by an ASTM D2PP statistical program. The significant results from these three round robin tests will be summarized in the following.

Cleaning Procedure

A survey of tribological test procedures revealed the use of a variety of different cleaning solvents. DIN 51834-2 requires single boiling spirit (sbp) according to DIN 51361-2-A (80°C to 110°C boiling range) and ASTM Test Method for Measuring Friction and Wear properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test machine (D5707). The ASTM Test Method for Determining Extreme Pressure Properties of Lubricating Grease Using a High-Frequency, Linear-Oscillation (SRV) Test machine (D5706) stipulates to use a mixture of equal volumes of n-heptane, toluene and isopropanol.

Table 3: *Ranking of Wear Scar Diameter and COF by Using Different Cleaning Solvents*

Test oils CEC RL 175/3 and PAG 50-100B
COF smaller <Isopropanol/Toluene/Hexane<Acetone<single boiling point spirit < Petrolether <Isohexane <Isoparaffin < COF greater
Wear scar smaller < Isopropanol/Toluene/Hexane, Petrolether< Single boiling point spirit< Isoparaffin, Isohexane < Wear scar greater
Test oils ISO VG 40 and ISO VG 220 (additivated paraffinic oils)

Table 4: *Correlation between the model and the mean of the COF of different models in the RR98*

Interfacial media	Number of COF-end	Mean of COF-end	Model of Tribometer
Test oil A	4	0.1110	ICC
Test oil B	4	0.1352	ICC
Test oil A	2	0.1360	SRV 0
Test oil B	2	0.1535	SRV 0
Test oil A	18	0.1149	SRV I
Test oil B	18	0.1424	SRV I
Test oil A	8	0.1145	SRV II
Test oil B	8	0.1387	SRV II
Test oil A	14	0.1232	SRV III
Test oil B	14	0.1500	SRV III

Ultrasonic cleaning was reported by 85% of the participants. The participants in the RR tests used more than 10 different cleaning solvents. The cleaning solvent must influence the test results because the steel samples are protected against corrosion. In consequence, the remaining layer of the rust inhibitor depends on the cleaning effect of the solvent. Table 3 compiles the ranking of results by cleaning solvents observed as clusters in the RR97 and RR98. It is evident that the friction and wear data are ranked by the type of cleaning solvent used.

Model of Test Equipment

In the last few years, the performance of the test machine was continuously improved. Three machine generations have been developed which vary with regard to the electronics (data acquisition, control unit) and design (maximum stroke, normal force). The maximum load for the SRV I is 1,200 N, for the SRV II it is 1,400 N and for the SRV III 2,000 N. The question was: does the model (SRV I, SRV II, SRV III and ICC) influence the tribological results?

Tables 4 and 5 rank correlate the different types of machines with the coefficient of friction and wear scar diameter for two oils. On the basis of a COF= 0.1, the model SRVIII report COFs which are 0.7% to 0.9% higher than those of SRVI/II-models. SRVI models report COFs which are 0.18% to 0.36% higher than those of SRVII machines.
No clusters were observed between the models with respect to wear scar diameters. A similar ranking was found in the RR97 and RR99.

Table 5: *Correlation between the model and the mean wear scar diameter of different models in the RR98*

Interfacial media	Number of W_K	Mean of W_K	Model of Tribometer
Test oil A	4	0.59350	ICC
Test oil B	4	0.70000	ICC
Test oil A	2	0.70000	SRV 0
Test oil B	2	0.72500	SRV 0
Test oil A	18	0.54809	SRV I
Test oil B	18	0.72668	SRV I
Test oil A	8	0.50812	SRV II
Test oil B	8	0.64125	SRV II
Test oil A	14	0.55278	SRV III
Test oil B	14	0.70014	SRV III

Wear Scar Diameter

With 0.478 mm (P_H wear = 1,665 MPa), the value of the optically visible average wear scar diameter for the ball tested with Mobil SHC is slightly above the average Hertzian diameter of 0.427 mm using F_N=300 N (corresponding to an initial average Hertzian pressure of P_0= 2,092 MPa). All participants stated for this oil a wear scar diameter as wear amount, because they saw a colored scar in the optical microscope.

Figure 2 shows only a tiny difference in the stylus traces of the unstressed surface and the wear scar. In order to rank and better discriminate between different fluids, Table 6 illuminates the importance of the use of the wear volume and the difference in using an equation and stylus profilometry [*7,12*].

Table 6: *Comparison between the values of wear scar diameter and wear volume on one ball*

Wear quantity ball	MOBIL I SHC 500 VG 46	KAJO-BIO-HEES-S VG 46
Scar diameter [mm]	0.478000	0.79100 (times 1,65)
Wear volume from equations in DIN 51834, part 3 [mm³]	0.000164	0.00122 (times 7,4)
Wear volume from stylus profilometry [mm³]	~0.000035	~0.0008 (times 22,8)

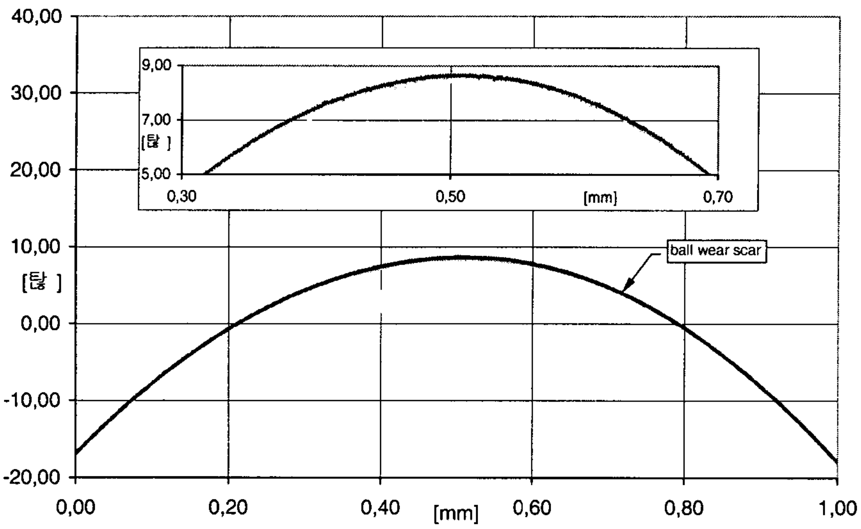


Figure 2: *Stylus profilometry across the center of the ball wear scar (stylus TK300)*

The equation in DIN 51834, part 3, assumes that wear occurs on both samples and the wear volume of the ball can be calculated from the planimetric wear [μm^2] of the track perpendicular to the sliding direction. Testing the MOBIL hydraulic oil, some wear on the track (flat sample) was detectable by means of stylus profilometry, but in this particular case, the ball grooved into the disk, so the equations can't be applied as shown in Table 6. The wear volume on this ball is the difference between the trace of the unstressed surface and the wear scar in Figure 2.

Seizure Criteria

The RR99 showed for the saturated synthetic ester hydraulic oil and the unadditivated PAG several sharp rises (short peaks) in COF in the testing period 5-10 minutes to 15-20 minutes. After this period of, say running-in, the COF (see Figure 3) decreased continuously and behaved smoothly. The optical inspection of the scar and track didn't reveal any evidence of adhesive transfer. According to the criteria defined in section "9.2.2" in DIN 51834-2:1997, ~20% of the participants stopped the test and stated "adhesive failure".

Similar frictional behavior was observed for the ester based biodegradable, low additive hydraulic/transmission lubricant [*13*]. The development of peaks depends on the Hertzian pressure. Peaks appear at a Hertzian pressure above 1.5 GPa. This ester tested in the RR99 meets the technical specifications of construction equipment manufacturers, but would be rated "non-working" using the seizure criteria in DIN 51834-2:1997.

The extreme pressure characteristics using D5706 [*14*] are evaluated by changes in friction characteristics, whereas EP lubricants, while often (but not always) providing friction modification, are used primarily to control wear or surface damage. The seizure criteria of DIN 51834-2 are also based on the changes in the friction characteristics. It would seem that a test for EP characteristics of a lubricant should include the evaluation of both friction and wear (and/or surface damage) of the test pieces at a defined Hertzian pressure, which should be reported with the tribological results (see also Chapter seizure).

The coefficient of friction of self-mated AISI 52100 (100Cr6H) couples lies between 0.5 and 0.9 at room temperature and under dry conditions (without adhesive failure, but strong tribooxidation) [*15*].

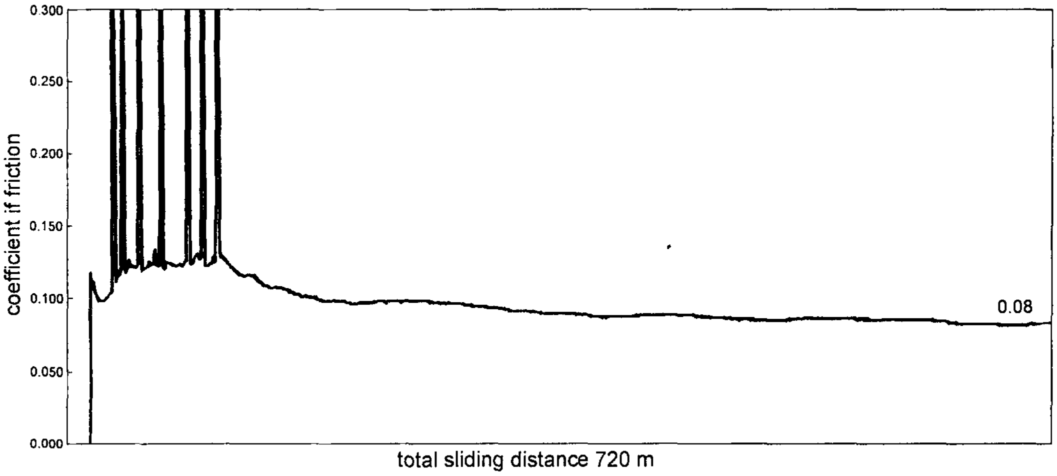


Figure 3: *Typical trace of COF versus sliding distance of the saturated, synthetic ester during RR99 (T= 50°C, F_N= 300N, Δx= 1 mm, v= 50 Hz, t= 120 min)*

We have observed in the RR tests, because 80% of the participants didn't apply the seizure criteria as defined in DIN 51834-2:1997, that both the PAGs and the ester exhibited low friction coefficients and running-in properties without any indications of seizure by visual inspection of the wear scars, when the Hertzian pressures was below ~1.500 MPa.

The DIN and ASTM working groups now elaborate typical graphs, where seizure occurs as an annex of the procedure.

Stroke

ASTM D5706-97 [9] uses a stroke of 1,0 mm for EP-testing of greases. The int. RR 2000 according to ASTM D5706, as well as one RR previously performed 1998 in the US [16], have produced repeatabilities and reproducibilities of the EP-test procedure with greases, which don't fit with the outstandingly high statistical "safety" of DIN 51834, ASTM D5707 and ASTM D6425.

The DIN working group decided at its annual meeting end of September 2000, to perform an „ad hoc" RR with an increased stroke of 1.5 mm, by using the same grease samples and a caliper for applying the grease.

Figure 4 shows clearly that a displacement of 1.0 mm results in an unacceptable dispersion of the results. This becomes clear when comparing the range of measurements of the wear scar diameter (between 0.75 mm to 1.4 mm) with the stroke of 1.0 mm. Using 1.0 mm of stroke, parts of the wear track are always in contact and can't be wetted with grease.

A survey of the labs who had participated proved that the requirement "place a small amount (0.1 to 0.2 g) of lubricating greases to be tested on the cleaned disk" can be misunderstood. The application of a grease caliper solves this problem.

Figure 4 compares the failure loads registered under 1 mm stroke to those under 1.5 mm stroke. Due to the different maximum loads of the models the results obtained on ICC/SRVI/SRVII and SRVIII exhibit differences.

It was observed that the models ICC/SRVI/SRVII all reach 1200 N without adhesive failure taking place. This applies for both greases and a stroke of 1.5 mm. With 16 participants and repeated tests, the data have a limited significance.

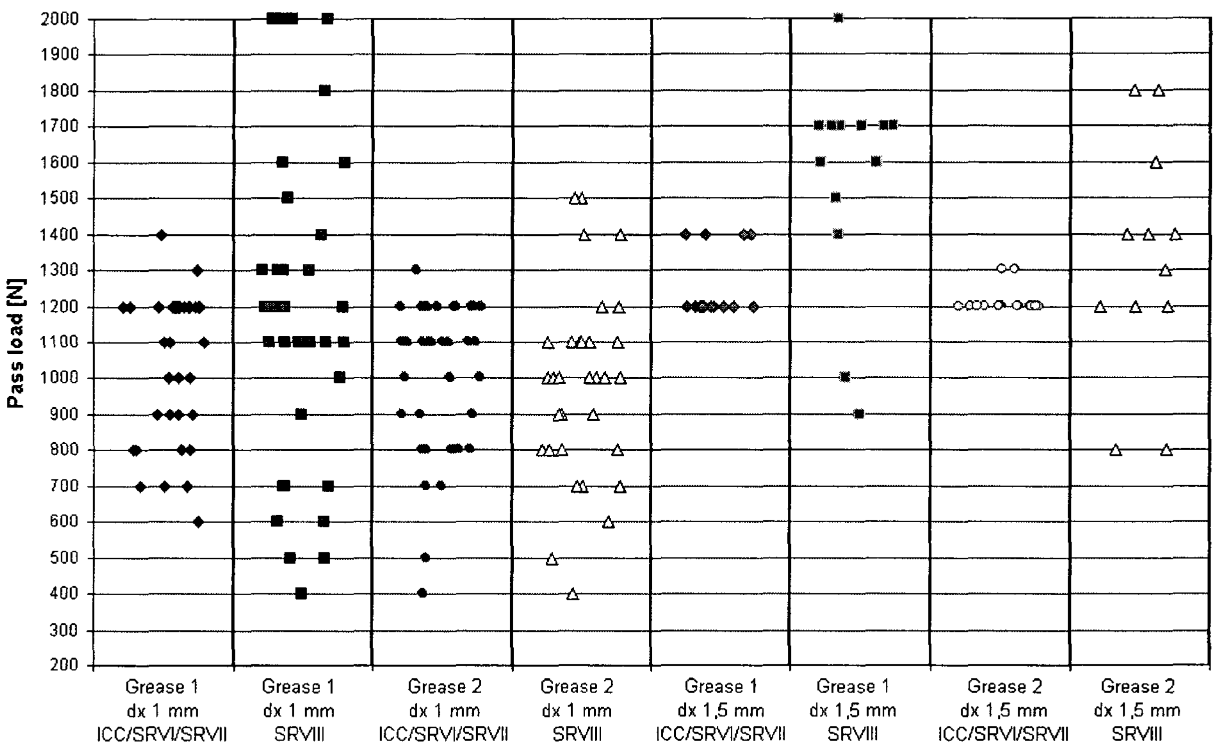


Figure 4: Pass load according to ASTM D5706 of grease 1 (Fina Ceran LT) and grease 2 (ASTM G-2000-02 Lithium Complex Grease with MoS_2, Zn/S/P) under two different strokes

Table 7: Mean of pass load according to ASTM D5706 including outliers in the „ad hoc" RR2000

Grease	RR2000 1 mm stroke (37-41 part.)	"ad hoc" RR2000/2001 1.5 mm stroke (16 part.)
	Mean value	Mean value
Fina Ceran LT (all results)	1098+/-395 (75 results)	1387+/-252 N (32 results)
ASTM G-2000-02 Lithium Complex Grease with MoS_2, Zn/S/P (all results)	997+/-235 (71 results)	1254+/-198 N (32 results)
Fina Ceran LT (adhesive failure reported)	1120+/-247 N (37 results)	**1560**+/-280 N (15 results)
ASTM G-2000-02 Lithium Complex Grease with MoS_2,	1036+/-228 N (41 results)	**1278**+/-235 N (23 results)

Zn/S/P (adhesive failure reported)		

Table 7 summarizes the mean value with data coming from all models and participants (including outliers). The occurrence or non-occurrence of adhesive failure was not taken into account. In the same way, the occurrence of adhesive failure at high loads was not taken into account.

From a statistical point of view, it is visible, that the mean high load (as pass load with adhesive failure) using 1,5 mm stroke can class the two greases in a safe way. With the outliers, the standard deviation is still high.

The number of results (1,5 mm) is smaller than 32, because adhesive failure must occur in order to consider the individual result.
For grease (ASTM G-2000-02 Lithium Complex Grease with MoS_2, Zn/S/P) , the machines SRVI/SRVII/ICC limited to 1.200 N achieved 10 results with no adhesive failure and 8 adhesive failures !
With a failure load of ~1.560 N, the machines ICC/SRVI/SRVII can't observe adhesive failure due to the limit of 1.200 N, so they have no results for grease " Fina Ceran LT ".

These results justify the increase of the load capacity of the SRVI/SRVII/ICC-machines to 1.600 N as the DIN 51834 working group had decided in its 29th September 2000 meeting.

Table 8: Passload of different greases under two different strokes (1 mm stroke for RR 2000; 1,5 mm stroke for RR 2001 and the use of a grease caliper) according to ASTM D5706

Year	**RR2001**	**RR2001**	**RR2000**	**RR2000**
Test greases D5706	**Li/Ca-12-OH-Stearat** **Δx= 1,5 mm**	**PAO/Polybuten/ Bentonit** **Δx= 1,5 mm**	**Fina Ceran LT,** Calcium-Sulfo-nate-complex **Δx= 1 mm**	**ASTM G-2000-02** Lithium complex with MoS_2, Zn/S/P **Δx= 1 mm**
Statistical Quantities (DIN ISO 5725, 4259)	Highest test load [N]	Highest test load [N]	Highest test load [N]	Highest test load [N]
Degree of freedom	30	39	39	53
Mean	**1.028**	**505**	**1.119**	**989**
Standard deviation	**+/-217**	**+/-106**	**+/-361**	**+/-260**
Reproduci-bility **R** (DINISO4259)	627	303	1.033	739
Repeatabi-lity **r** (DINISO4259)	219	197	239	455

The positive influence of the increased stroke on the standard deviation, reproducibility and repeatability is clearly visible in Table 8. The caliper for applying grease is also recommended to ensure the correct amount of grease in the contact.

The last remaining parameter in order to increase the repeatability and reproducibility is the teaching of the operators to distinguish between the pass load and failure load!
Pre-tests have shown that the increase of the running-in period additionally improves precision.

Table 9 : Pass load of different greases using Δx= 1,5 mm stroke at 80°C according to procedure "B." in ASTM D5706

Year	RR2003	RR2002	RR2001	RR2003	RR2002	RR2001
Test greases D5706	**Li/Ca-12-OH-Stearat**	**Li/Ca-12-OH-Stearat**	**Li/Ca-12-OH-Stearat**	**PAO Polybuten/-bentonit**	**PAO Polybuten/-bentonit**	**PAO Polybuten/ Bentonit**
Modifica-tions	Δx= 1.5 mm, grease apply caliper, O.K.-load	Δx= 1.5 mm, grease apply caliper	Δx= 1.5 mm	Δx= 1.5 mm, grease apply caliper, O.K.-load	Δx= 1.5 mm, grease apply caliper	Δx= 1.5 mm
Statistical Quantities	Highest test load [N]	Highest test load [N]	Highest test load [N]	Highest test load [N]	Highest test load [N]	Highest test load [N]
Degree of freedom	17*	28	30	32	27	39
Mean	**1.109$^+$**	**1.180**	**1.028**	**430$^+$**	**486**	**505**
Standard deviation	**+/-246**	**+/-235**	**+/-217**	**+/-71,3**	**+/-129**	**+/-106**
Reproduci-bility **R**	735	680	627	205	374	303
Repeatabi-lity **r**	285	267	219	179	201	197

NOTE: The repeatability and the reproducibility were calculated using the ASTM software „D2PP". * preliminary results before working group meeting, (16 results will be added later), $^+$ increased running-in

Table 9 shows, that precision could be improved for the PAO Polybuten-bentonit grease. The working group has decided on 26.09.2003 to carry out the 2004 RR with a grease having an O.K.-load of 900-1,000 N so that SRV I models can observe seizure.

The data of the Li/Ca-12-OH-Stearat grease contain results from SRVI-models without seizure or in other words: if seizure occurred it can be stated, since the maximum load of 1,200 N was not reached.

Greases versus oils

The different procedures characterize the tribological properties of solid bonded films (DIN 51834-8 draft), oils (ASTM D6495 and DIN 51834-2/-3) and greases (ASTM D5707). Since greases have a much lower flow behavior than oils, there is the question whether they can penetrate into the contact while testing at 1.0 mm stroke.
By virtue of the data from the RR tests, this question can easily be answered: Table 10 compares the statistical data of friction and wear tests according to D5707 for greases using F_N= 200 N and for oils using F_N = 300 N according to D6425/DIN51384-2.

Since the set points or limits for the reproducibility of greases and oils are identical or close together and as the repeatability for the coefficient of friction of greases is less than half that of oils, the statistical results can be considered and compared. Table 10 shows no evidence for an influence of the "flow" behaviour using 200 N or 300 N when characterising the friction and wear behaviour of oils and greases.

Discussions Within the Working Group

The actual versions of DIN 51834:1997 are valid until April 2002, and the corresponding ASTM D6425 have been valid since March 2003. Based on three round-robin tests and some round robin tests performed by companies owning five to seven test machines, a discussion on DIN 51834, part 2, regarding the procedure and the following modifications was accomplished by September 2000 and published in December 2001 as "yellow print". Most of these modifications appeared in the modified edition of ASTM D6425-02.

Table 10 Comparison of the repeatability and reproducibility of greases and oils according to DIN EN ISO 4259 and using the software ASTM „D2PP". " without outliers and >32 individual results for RR2000 and RR2001

Year	**RR2001**		**RR2001**		**RR2001**		**RR2000**	
Greases (D5707)	**Li/Ca-12-OH Stearat grease**		**PAO/Polybuten/ Bentonit**		**SHELL Omala oil HD 220**		**BP Energol GR-XP-220**	
Statistical quantity (DIN ISO 5725, 4259)	Coefficient of friction f_{end}	Wear scar diameter of the ball $W_{K\ average}$ [mm]	Coefficient of friction f_{end}	Wear scar diameter of the ball $W_{K-average}$ [mm]	Coefficient of friction f_{end}	Wear scar diameter of the ball $W_{K-average}$ [mm]	Coefficient of friction f_{end}	Wear scar diameter of the ball $W_{K-average}$ [mm]
Degree of freedom	33	35	33	31	33	34	39	45
Mean	**0.1175**	**0.5071**	**0.1162**	**0.5402**	**0.1006**	**0.4683**	**0.1105**	**0.47397**
Deviation σ	± 0.0152	± 0.0284	± 0.0138	± 0.0387	± 0.0128	± 0.0216	± 0.00891	± 0.04030
Reproduci-bility **R** (DINISO4259)	0.02 (limit: ≤0.032)	0.0817 (limit: ≤0.24)	0.0398 (limit: ≤0.032)	0.1116 (limit: ≤0.24)	0.0369 (limit: ≤0.04)	0.062 (limit: ≤0.20)	0.02540 (limit: ≤0.04)	0.1146 (limit: ≤0.20)
Repeatability **r** (DINISO4259)	**0.0107** (limit: ≤0.008)	0.0311 (limit: ≤0.07)	0.00854 (limit: ≤0.008)	0.0246 (limit: ≤0.07)	0.00873 (limit: ≤0.02)	0.0266 (limit: ≤0.1)	0.00772 (limit: ≤0.02)	0.06680 (limit: ≤0.1)

Wear Volume

The wear volume will replace the wear scar diameter (optional in the future ASTM D6425-02), but the wear scar diameter is still valid. This was already introduced into DIN 51834, part 3. If the wear scar diameter is smaller than 1.2 times the average Hertzian contact diameter, then the wear loss has to be proven with profilometry and the wear volume has to be calculated from the stylus trace, because the wear scar is close to the Hertzian diameter. A measurable (optically visible) wear scar diameter may or may not indicate wear on the surface of the ball and the wear track (see chapter wear scar diameter).

Additionally, in DIN 51834, part 2 and part 3, the geometric pressure at test end will be reported.

Seizure

The COF value of f>0.35 will be cancelled. Any peak over delta 0.2 is an undesired event for lubricated contacts. The seizure criteria for lubricated contacts will be a sharp rise of delta 0.2 over 30 seconds or the proof of adhesive transfer in SEM or light-optical microscopic pictures. Similar findings were made in the development of the draft for DIN E 51834-8.

In order to illuminate the different cases in which seizure may or may not have occurred, typical friction graphs will be added to the grease standards.

Temperature

Especially the automotive industry expressed the wish to test at higher temperature. Tests at a constant temperature of 90°C and 135°C will be run in addition to the test at 50°C.

Oil Volume

According to systematic research of some customers the oil volume will be increased to 0.3 ml.

Load

The constant test load can be chosen from any load in 100 N steps. This flexibility is to allow to determine the tribological quantities of friction and wear at an appropriate load where no seizure occurs. The round robin tests performed with greases by US customers (97-98) and in the int. RR20001 revealed that a normal force of F_N= 1,200 N is not enough for some EP-greases to observe seizure. The normal force required in DIN 51834, part 1, " General principles..." will be increased from 1,200 N to 1,600 N. For SRVI and II models, an up-grade kit to 1,600 N is available.

Coefficient of Friction

The coefficient of friction value will be indicated to the third decimal point.

Topography of Specimens

The surface of the lapped flat samples will be defined as follows: 0.035 μm < R_a (C.L.A.) < 0.05 μm, 0.02 μm<R_{pk} < 0.035 μm, 0.05 μm< R_{vk} < 0.074 μm and 0.5 μm< R_z < 0.65 μm. The topography needs to be precisely specified in order to facilitate the manufacture of specimens which conform with the requirements of the standard. R_{vk} and R_{pk} are according to DIN EN ISO 13565-2:1998.

The surfaces of the disk are lapped and free of lapping raw materials.

Stroke

The stroke will be checked periodically by subtracting the wear scar diameter of the ball from the wear track length which must be identical with the selected stroke.

Flat Samples

The RR showed that the results of a customer who had machined his own test specimens in accordance with DIN 51834-2 lie within the statistical limits. Surface analysis of the specimens showed that stragglers and outliers correlated with the presence of lapping grits in the grooves of the roughness valleys. For future tests, the surfaces of test specimens are required to be free from other lapping grits. Four roughness values now characterize the surface topography.

Precision and bias

The most important changes are related to the precision statements. They reflect the improvement in running the procedure for the operators. The systematic errors were continuously reduced over the years. The RR test campaigns 1997 to 2003 have produced reproducibility and repeatability values which are in some cases one third or one fourth of the set points (limits) stated in DIN 51834-2:1997 or ASTM D6425-99.
The precision statement in a standard affects the commercial relationship between the supplier and the user of lubricating products. This context has caused the DIN working group to adopt only a small improvement or no changes in the precision statements even though more would have been justified on the basis of the statistical data. On the other hand, however, the following note will appear in chapter 11 of the new edition ASTM D6425 and DIN 51834-2:2002:

"The repeatability and reproducibility values depend on the oil that has been used. The values stated in sections 11.2.1.1. to 11.2.2.2. represent maximum values. In singular cases, smaller values were determined in RR tests according to DIN 51834, part 2."

The Table 11 compares the precision statement of the edition ASTM D6425-99 or DIN 51834-2:1997 in parentheses with the new values in bold of the next editions which will appear in DIN 51834-2:2002 and the future D6425-02. The precision statements of the SRV grease and oil test procedures are now nearly identical.

Table 11: Comparison of the values for the precision statements for F_N= 300 N, T= 50 °C and 0.03 ml

Quantity	**Repeatability**	**Reproducibility**
Coefficient of friction at test end **f**	**0.01** (0.02)	**0.03** (0.04)
Wear scar diameter ball **Wk** in mm	**0.07** (0.1)	**0.2** (0.2)

The statistical results for oils in the RR2000 and RR2001 (see Tables 10, 12 and 13) are clearly also within the new limits.
The unsatisfactory point in the RR test campaign 1997 to 2003 is a degree of freedom much lower than the number of single results, even the number of single results ranges from 45 to 72. The reason lies in the report of the identical individual results of one lab code, which are considered as one result instead of two!

Influence of temperature

The new approved ASTM D6425 and DIN 51834-2 allow tests at temperatures higher than 50°C. The aim of the RR 2002 and 2003 was to establish the precision. The BP Energol

GR-XP-220 and SHELL Omala oil HD 220 were tested at 80°C and 120°C in order to compare the results with those from 50°C.

Tables 12 and 13 show that precision increases with increasing temperature.

Report

The tribological quantities determined have to refer to the operating conditions as follows: DIN51834-2/normal force/temperature or for example DIN51834-2/600/90.

Table 12: Statistical analysis of the tribological quantities (7th Int. Round Robin Test 2003 ASTM D6425/DIN 51834-2, preliminary)

Year	**RR2003**		**RR2003**		**RR2000**	
Test oil	**BP Energol GR-XP 220**					
Temperature	**120°C**		**80°C**		**50°C**	
	Tribological Quantities					
Statistical Quantities (D2PP)	Coefficient of friction f_{END}	Wear scar diameter of the ball $W_{K\text{-}average}$ [mm]	Coefficient of friction f_{END}	Wear scar diameter of the ball $W_{K\text{-}average}$ [mm]	Coefficient of friction f_{END}	Wear scar diameter of the ball $W_{K\text{-}average}$ [mm]
Degree of freedom	19	20	19	19	39	45
Mean	**0.0975**	**0.4801**	**0.1030**	**0.4708**	**0.1105**	**0.4739 7**
Standard deviation	± 0.00803	± 0.0193	± 0.00699	± 0.0211	± 0.00891	± 0.04030
Reproduci-bility **R**	0.0238 (set point: ≤0.03)	0.0569 (set point: ≤0.2)	0.0207 (set point: ≤0.03)	0.0625 (set point: ≤0.2)	0.02540 (set point: ≤0.,03)	0.1146 (set point: ≤0.2)
Repeatability **r**	0.0042 (set point: ≤0.01)	0.0274 (set point: ≤0.07)	0.00469 (set point: ≤0.01)	0.0203 (set point: ≤0.07)	0.00772 (set point: ≤0.01)	0.06680 (set point: ≤0.07)

Statistical analysis using ASTM „D2PP" software >55 single results per test oil. (Test conditions according to DIN 51834, part 2: F_N= 300 N; t= 120 min; **Δx= 1 mm**; ν= 50 Hz; lapped AISI 52100-disks; polished AISI 52100-balls,∅= 10 mm)

Table 13: Statistical analysis of the tribological quantities(6th Int. Round Robin Test 2002 ASTM D6425/DIN 51834-2)

Year	**RR2002**		**RR2002**		**RR2001**	
Tes toil	**SHELL Omala Oil HD 220**					
Temperature	**120°C**		**80°C**		**50°C**	
	Tribological Quantities					
Statistical Quantities (D2PP)	Coefficient of friction f_{END}	Wear scar diameter of the ball $W_{K\text{-}average}$ [mm]	Coefficient of friction f_{END}	Wear scar diameter of the ball $W_{K\text{-}average}$ [mm]	Coefficient of friction f_{END}	Wear scar diameter of the ball $W_{K\text{-}average}$ [mm]
Degree of freedom	31	29	29	26	33	34
Mean	**0.0895**	**0.4824**	**0.0919**	**0.477**	**0.1006**	**0.4683**

Standard deviation	0.0095	0.0226	0.00701	0.0261	± 0.0128	± 0.0216
Reproduci-bility **R**	0.0287 (set point: ≤0.03)	0.0654 (set point: ≤0.2)	0.0203 (set point: ≤0.03)	0.00758 (set point: ≤0.2)	0.0369 (set point: ≤0.03)	0.062 (set point: ≤0.2)
Repeatability **r**	**0.00132** (set point: ≤0.01)	0.035 (set point: ≤0.07)	0.00778 (set point: ≤0.01)	0.026 (set point: ≤0.07)	0.00873 (set point: ≤0.01)	0.0266 (set point: ≤0.07)

Statistical analysis using ASTM „D2PP" software >55 single results per test oil. (Test conditions according to DIN 51834, part 2: F_N= 300 N;t= 120 min; **Δx= 1 mm**; ν= 50 Hz; lapped AISI 52100-disks; polished AISI 52100-balls,Ø= 10 mm)

Conclusions

Round robin tests represent a versatile instrument to identify and quantify systematic and other errors of the test procedure and are an excellent training for users. They increase the knowledge on the influence of test parameters and/or operation conditions on the tribological quantities. They are also needed to prove regularly that machine and operator produce credible results in order to respond to the demand for a highly reproducible and repeatable test equipment and procedure. The round robin data shows the ranking of the COF by cleaning solvents and that a measurable (optically visible) wear scar diameter may or may not indicate wear on the surface of the ball and the wear track. The wear volume determined by stylus profilometry represents the safest way to report wear. Also the RR have displayed the influence of stroke, temperature, running-in and oil volume on the tribological results. The seizure criteria have to reflect the tribological performance of products.

Acknowledgements

The authors would like to thank the DIN Working Group 51834, the ASTM D02.L sub-committee and the SRV users for conducting the RR tests and the invaluable discussions. This paper could not have been written without the experimental help of more than 70 different laboratories worldwide.

References

[1] Van de Velde, P. Willen, and Van Geetruyen
Substitution of inexpensive bench test for the FZG scuffing,
part I : calculations, Tribology Transactions, Vol. 42, No. 1, 1999, p, 63-70
part II: Oil tests, Tribology Transactions , Vol. 42, No. 1, 1999, p. 71-75

[2] DIN 51834-1 General principles for the tribological testing of lubricants using a linear-oscillation test machines, April 1997, Beuth Verlag, D-10772 Berlin

[3] DIN 51834-2 Determination of friction and wear characteristics for lubricating oils using a linear-oscillation test machine, April 1997, Beuth Verlag, D-10772 Berlin

[4] Dickey, J.
New ASTM and DIN methods for measuring tribological properties using SRV test instrument
NLGI Spokesman, March 1997, p. 17-23

[5] J. Flörchinger
Schwingreibverschleissprüfung für Schmierstoffe (SRV-Test) - Tribologische Untersuchung von Schmierfetten für Gelenke mit Kunststoff-Gelenkschale.
TRW Fahrwerkssysteme 62 051 301

[6] Nieddu
Entwicklungshandbuch Lastenheft - Gleitaktive Multifunktionsbeschichtungen
Kiekert 4907 0038

[7] DIN 51834-3 Testing of lubricants – tribological test in translatory oscillation apparatus- Part 3: Determination of tribological behavior of materials in co-operation with lubricants, white print in press, Beuth Verlag, D-10772 Berlin

[8] Klaffke, D.,"On the Influence of Some Test Parameters on the Results of Oscillating Sliding Tests", Tribotest Journal 6-1, 1999, p. 29-49

[9] Woydt, M., „Ergebnisse des Ringversuches zur DIN 51834", Tribologie&Schmierungstechnik, 44. Jg, Heft 6, 1997, p. 284

[10] Woydt, M., „Einfluß der Reinigungsmedien auf die Prüfung tribologischer Kenngrößen", Tribologie&Schmierungstechnik, 46. Jg, Heft 2, 1999, p. 36

[11] Woydt, M.
Influence of test parameters on tribological results – Synthesis from round robin tests
In: Bench Testing of Industrial Fluid Lubrication and Wear Properties Used in Machinery Applications, ASTM STP 1404, G. E. Totten, L. D. Wedeven, J. R. Dickey, M. Anderson, Eds., American Society for Testing and Materials, West Conshohocken, PA , 2001, p.199-209, ISBN 0-8031-2867-3

[12] Kalin, M. and J. Vižintin, "Use of Equations for Wear Volume Determination in Fretting Experiments", WEAR, Vol. 237, 2000. P. 39-48

[13] Vižintin, J. and A. Arnsek, "Scuffing Load Capacity of Rapeseed-Based Oils", Lubrication Engineering, August 1999, p. 11-19

[14] ASTM D5706-98 "Determining Extreme Pressure Properties of Lubricating Greases Using a High-Frequency, Linear-Oscillating (SRV) Test Machine", ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428, USA

[15] Klaffke, D., „On the Repeatability of Friction and Wear Results and On the Influence of Humidity in Oscillating Sliding Tests of Steel-Steel Pairings", WEAR, vol. 189, 1995, p. 117-121

[16] Dickey. J.
ASTM D 5706 SRV EP Test Interlaboratory Test Programme 1997-1998
ASTM D02.G0.04.02 Task Force on SRV Methods, g4rr-report, 18. November 1998