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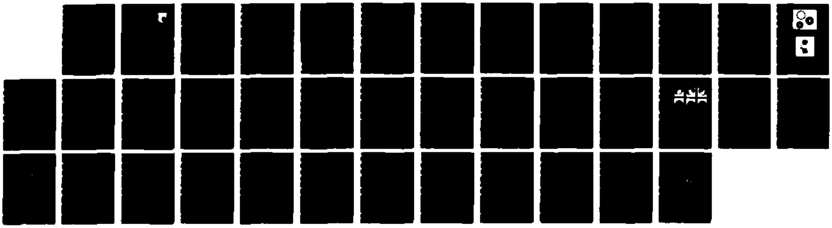

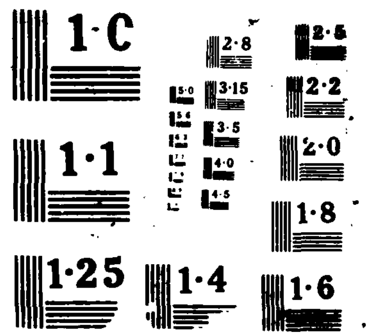
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STANDARDIZATION OF LUBRICITY TEST

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NOTICE

This technical report has been reviewed and is approved for publication.

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The technical effort described herein was directed at refining and standardizing the Ball-On-Cylinder Lubricity Evaluator (BOCLE) test. The thrust of the effort focused on identifying variables suspected of affecting test method precision. Recommendations based on the conclusions of this study were submitted to the Air Force Project Engineer for consideration and review by the Coordinating Research Council (CRC) BOCLE Operators' Task Force and Fuel Lubricity Panel.

The Falex Ring was shown to significantly enhance BOCLE test precision and eliminate many of the problems associated with the conventional AMS 6444 test cylinder. Test results showed that the source of the test ball can have a significant effect on BOCLE test results. SKF Swedish precision balls were found to provide optimum batch to batch consistency. Isopar M + 30 ppm DuPont DCI-4A was shown to be a reproducible fluid suitable for use as a standard reference fluid for BOCLE testing.

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FOREWORD

This report describes the technical effort conducted under Air Force Contract No. F33615-85-C-2508, entitled "Properties of Aircraft Fuels and Related Materials." All research conducted under this contract was administered under the direction of Mr. Timothy L. Dues, Project Engineer, Fuels Branch of the Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratories and Mr. Paul A. Warner, Program Manager, United Technologies Corporation, Pratt & Whitney Engineering Division (P&W/ED).

The technical effort disclosed herein was performed during the period 30 November 1985 through 01 November 1986 under Task Order No. 3, entitled "Standardization of Lubricity Test." Tedd B. Biddle was the P&W Task Order Manager.

The authors wish to thank Timothy Dues, the Air Force Project Engineer, for his invaluable assistance in providing technical direction and support in the many decisions required for the successful completion of this program; and Buck Nowack and Jim Peluso of the Naval Air Propulsion Center (NAPC) for their counsel and interest in fuel lubricity. The authors wish to thank the following personnel of those laboratories, within industry and the military, which participated in the interlaboratory cooperative effort in the evaluation of the Falex Timken Ring: Tim Dues and Richard Striebich of AFWAL/POSF; Brian Rayner, Stuart Bullock and Chris Lewis of Rolls Royce, Ltd.; Robert McCarthy of Woodward Governor Co.; and Jim Clerc of Chevron Research Co. The authors also wish to acknowledge and thank the following P&W personnel: Michael Polito for his assistance and diligence in the Ball-On-Cylinder Lubricity Evaluator (BOCLE) analyses, which consisted in excess of 800 BOCLE runs; and Donald Yost for his assistance in maintaining sample inventory, and compiling and organizing of test data.

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SECTION I

INTRODUCTION

Lubricity may be the most critical fuel property likely to be degraded by refining methods. Future fuels will be refined from high sulfur and high aromatic crudes, as well as from shale oil and coal syncrudes with equally high sulfur and aromatic concentrations. Low quality feedstocks will necessitate the use of severe hydrotreating. This refinery process removes or reduces the lubricity enhancing polar molecules that naturally occur in crudes and provide the boundary lubrication necessary for engine fuel system components.

Lubricity related fuel system component problems first surfaced in the early 1960's. Catastrophic failure and extreme reduction in component life have been associated with low lubricity fuels of all types used by both military and commercial aircraft. Typical problems include severe bore wear, ball joint wear, and complete piston failure of piston type pumps. Gear type fuel pumps operating on low lubricity fuels have encountered journal bearing seizure, drive shaft failure, wear of gear teeth flanks, and flaking of the contact area of the teeth in the main stage drive gear. Most recently, incidents of F-111 aircraft hydraulic pump housing fractures have been reported by Cannon AFB, New Mexico; Plattsburgh AFB, New York; and Tinker AFB, Oklahoma.

A significant level of effort has been expended in the study of low lubricity fuels: chemical properties, lubricity enhancing additives, and test method development. Since the early 1960's, the Ball-On-Cylinder Lubricity Evaluator (BOCLE) has been recognized as the best available method for providing a relative system of measurement of the lubricity properties of jet fuels. A variety of ball-on-cylinder machines, test procedures, test cylinders, and reference fluids exist in the military and throughout industry. This lack of standardization serves to severely restrict test repeatability and reproducibility of data among different laboratories.

Standardization of the BOCLE test method is of fundamental importance in order that conventional and experimental fuels can be accurately characterized to predict potential problems or to determine the cause and mechanism of lubricity-related fuel system failures. Of equal importance is the ability to compare and interpret data from various laboratories. This can be accomplished only if test apparatus and procedures are standardized and variables affecting precision are minimized by way of definitive specifications.

The technical effort described in the following sections was directed toward refining and standardizing the BOCLE test. The thrust of the effort focused on identifying variables suspected of reducing test method precision. Recommendations based on the conclusions of this study are herein submitted to the Air Force Project Engineer for consideration and review by the Coordinating Research Council (CRC) BOCLE Operators' Task Force and Fuel Lubricity Panel.

SECTION II

EXPERIMENTAL

In order to accomplish the goals set forth in this investigation, the technical approach followed a step-like progression in identifying the primary variables suspected of affecting BOCLE test precision. Once identified, these critical parameters were evaluated within the constraints of the operating conditions of the BOCLE test. Based on the assessment, optimum conditions and material specifications were defined.

Technical effort included determination of an appropriate standard reference fluid to permit test cylinder calibration and interlaboratory comparison of data. Cylinders, which were fabricated by different vendors to selected surface finishes, were evaluated. This was done to assess the effect of surface finish on test precision. These tests also served to appraise vendor quality control in providing a source for repeatable and reproducible test cylinders. An alternative test specimen to that of the problem-laden conventional test cylinder, currently used in the BOCLE test, was investigated. A further investigation focused on test ball to determine whether there was a measurable effect on test precision between ball manufacturers. Work was concluded with an interlaboratory evaluation of the Falex Timken Ring. This cooperative effort assessed the ability of the Falex Ring to meet the criteria required for a standard specification test, enhance test precision, and resolve existing problems associated with the conventional BOCLE cylinder. Five laboratories participated in the mini-round robin.

All BOCLE tests were performed on an InterAv BOC-100 lubricity tester. With the exception of the variable or material specimen under test, the test procedure throughout the course of the program closely adhered to the CRC recommended BOCLE test method outlined in Draft #9, "Standard Test Method for Measurement of Lubricity of Liquid Hydrocarbon Fuels By the Ball-On-Cylinder Lubricity Evaluator".

1. REFERENCE FLUID INVESTIGATION

Determination of appropriate or desired properties for a candidate reference fluid was made by surveying the chemical and physical properties of various hydrocarbon type fluids in a range of carbon numbers from C6 to C12. Storage stability, long term availability, and the level of fluid harshness were among the criteria examined. It was considered desirable that the degree of fluid harshness permit sensitivity to exceptionally high lubricity fuels. It was also desirable to retain the precision which is sometimes lost as a result of larger BOCLE wear scar diameters (WSD). Pure hydrocarbon fluids and formulations of hydrocarbon fluids were considered along with additive blends and "neat" fuels. The merit of multiple fluids for purposes of cylinder calibration, quantitative lubricity references, and contamination detection was also examined.

Among the pure hydrocarbon solvents considered was Isopar M which is produced by the Exxon Company and used extensively in industry as a calibration fluid. Isopar M is an odorless, relatively high boiling, narrow cut isoparaffinic solvent of high purity. It is synthesized by a catalytic process from petroleum fractions. A table of physical and chemical properties characterizing Isopar M is presented in Appendix A. Reference fluid candidates included straight chain hydrocarbons (C6 to C12); a cycloparaffin, cyclohexane; naphthalene derivatives, decalin and tetralin; neat Isopar M; Isopar M with additive (DuPont DCI-4A); and blends of Isopar M and tetralin.

Candidate reference fluids were selected and evaluated by way of a series of BOCLE tests. The test matrix consisted of three cylinders of known and proven good quality and three cylinders of known inferior quality. Determination of "good" and "bad" cylinder quality was based on P&W experience and current standards as defined by the existing BOCLE test procedure. All good cylinders were fabricated to specifications defined by CRC recommended guidelines: 4 to 9 microinch (µin.) surface finish, 20 to 22 Rockwell C (Rc) hardness, and AMS 6444 alloy. Cylinders determined to be of poor quality varied in surface finish, hardness, and alloy. These cylinders were fabricated from an AMS 6440 alloy. The surface finish of the cylinders varied from 10 to 40 µin. Hardness varied from 22 to 24 Rc.

The candidate reference fluids were run in quadruplicate on each of the three good and three bad cylinders. The initial test run on a cylinder was spaced 1.0 millimeter (mm) from the edge of the hub-side of the cylinder. Succeeding runs were spaced in increments of 0.75 mm. The candidate fluids were tested in an alternating sequence across the face of each cylinder.

Selection of a standard specification reference fluid was based on the following criteria:

- Repeatability across the cylinder running surface
- Reproducibility from cylinder to cylinder
- Differentiation between cylinders of known good quality and cylinders of questionable or inferior quality
- Maintainability of a constant lubricity value over extended periods of storage
- Storage stability and availability in future years

2. TEST CYLINDER SURFACE FINISH INVESTIGATION

The fluid selected as the most promising standard at the conclusion of the reference fluid investigation was used to provide a baseline for assessing and comparing cylinder variables, and for quantifying their effect on test repeatability and reproducibility. The effect of surface finish on test cylinder repeatability, both across the running surface and from cylinder to cylinder, was examined in three separate phases. These three phases of investigation were necessitated by anomalies encountered during the first and second phases of surface finish testing.

The effect of surface finish was determined for two different surface finishes which had been previously verified by profilometer measurements. The two different manufacturers supplying test cylinders were assigned the codes "J" and "FV," respectively . Surface finishes evaluated consisted of FV fabricated cylinders ground to a 4 to 9 µin. surface and J fabricated cylinders ground to a 16 to 22 µin. surface finish. Material alloy and cylinder hardness, also considered to be important cylinder variables, were held constant. The specified material was an AMS 6444 alloy, tempered to a Rockwell hardness of 20-22 Rc. Three cylinders of each surface finish were evaluated. BOCLE tests were performed in quadruplicate and included the following fluids: (1) Isopar M + 30 parts per million (ppm) DCI-4A, as the most promising candidate reference fluid; (2) JP-4, which is highly volatile and typically produces a relatively small wear scar; (3) clay-treated (CT) JP-4, which generates a relatively large wear scar; and (4) JP-7, which is of low volatility and typically produces a wear scar diameter between that of an as-received JP-4 and a CT JP-4.

3. FALEX RING INVESTIGATION

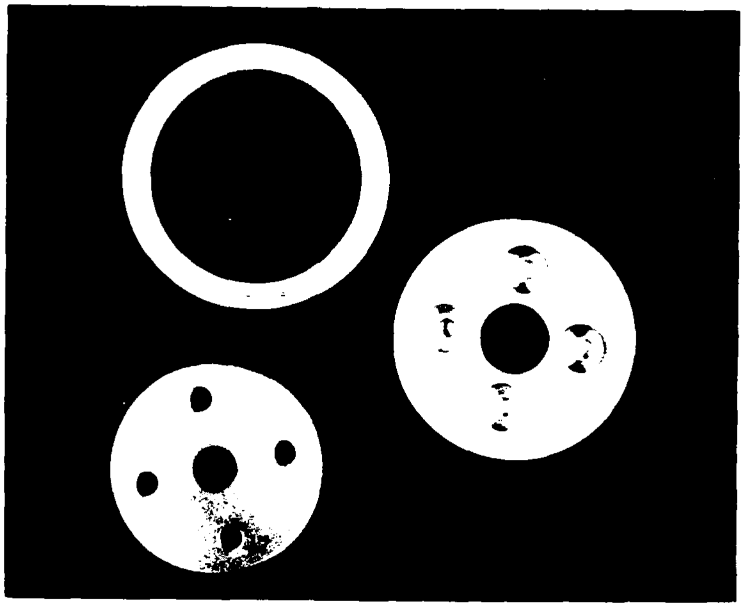
The Falex Ring is currently sanctioned by ASTM in two test methods which assess lubricating properties of oils (D2782 and D2509). The Falex Ring differs from the conventional solid BOCLE cylinder in alloy material, hardness, and surface finish. The alloy is an SAE 8720 modified steel, tempered to a hardness of 58 to 62 Rc, and ground to a 20-30 µin. surface finish. It has a 50 mm (1.97 inch) outside diameter (OD) and a 39 mm (1.54 inch) inside diameter (ID). The Falex Ring, fabricated by the Falex Corporation, is a readily available, low cost stock item.

Relatively minor special test conditions were necessary for use of the Falex Ring in place of the conventional AMS 6444 solid cylinder. A mandrel, also manufactured by the Falex Corporation, permitted installation of the Falex Ring on the standard InterAv cylinder shaft. Figure 1 shows the Falex Ring, mandrel, and the manner of installation. Because the Falex Ring is 5.55 mm (0.21 in.) larger in diameter than a conventional BOCLE cylinder, a minor modification to the BOCLE apparatus itself was necessary to permit leveling of the load arm. The modification consisted of inserting a 38.1 x 76.2 mm (1-1/2 X 3 in.) shim, made from a piece of 2.28 to 2.79 mm (0.090 to 0.110 in.) sheet metal shim stock, between the load pedestal and the top base plate. It was also necessary to attach a 19.0 X 19.0 mm (3/4 X 3/4 in.) shim to the underside of the load beam in such a manner that the hydraulic lift plunger met the shim when the plunger was fully extended in the "up" position. This was required to compensate for the plunger's limited length of travel. Appendix B contains a diagram with instructions to assist in modifying the BOCLE apparatus for use with Falex Rings.

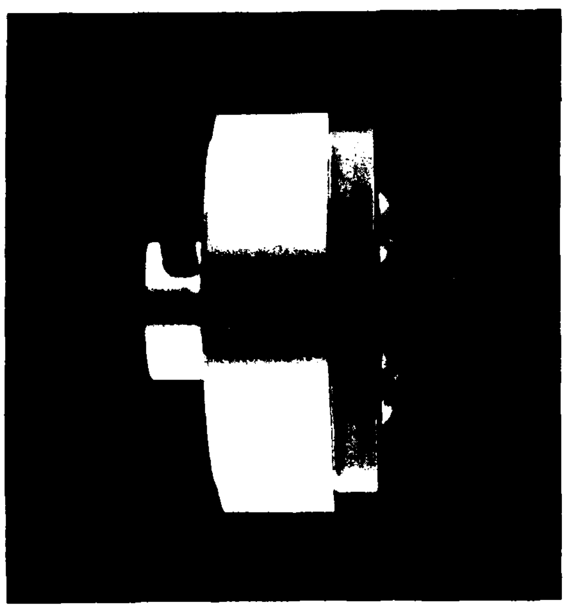
A reduction in applied load from 1000 grams (g) to 500 g represented the only change in the actual test conditions. The harder ring material was found to generate a larger wear scar than the conventional cylinder. After a series of trial runs with harsher test fluids, it was determined that a 500 g applied load was more suitable for maintaining a wear scar within the limits of the 1 mm graduated reticle of the microscope.

A preliminary investigation was performed to evaluate the potential of the Falex Ring to enhance BOCLE test precision. The test matrix consisted of three runs performed on each of three rings from the same material lot. Evaluations were conducted using five different fuel types. A total of 45 runs were performed.

As a result of the promise shown in the initial phase of the Falex Ring evaluation, a more extensive investigation was performed. This investigation consisted of 180 runs and was designed to determine repeatability of the Falex Ring in a variety of fuel types, degree of differentiation between fuel types of varying known lubricity, reproducibility of data from ring to ring within a given production lot, and reproducibility of data from the manufacturer's lot to lot production. Three rings were evaluated from each of four different production lots in five different fuel and fluid types. The test fluids and fuel types included the selected BOCLE reference fluid (Isopar M + 30 ppm DCI-4A), JP-4, JP-8, JP-7 and CT JP-4. Tests were performed in triplicate on each test ring. This generated a total of 36 data points for each fuel sample.



(a) Falex Ring, Right and Left Halves of Mandrel Assembly



(b) Assembled Falex Ring and Mandrel

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Figure 1. — Falex Ring and Mandrel Assembly

4. INTERLABORATORY EVALUATION OF THE FALEX RING

An interlaboratory cooperative effort was organized to confirm the potential shown by the Falex Ring to enhance test precision. The intent of the mini-round robin was to generate a preliminary precision statement before recommending a full-scale round robin sponsored by the CRC. Five laboratories participated in the cooperative effort. A summary sheet was forwarded to each facility outlining the objectives, special test conditions, test matrix, and special instructions for the performance of the test program.

The test matrix was designed to evaluate the Falex Ring on the basis of the following criteria:

- Differentiation between fuel types
- Repeatability across the running surface
- Repeatability from ring to ring
- Reproducibility from lot to lot
- Objectivity of wear scar interpretation

All necessary hardware was supplied to the participating independent laboratories by P&W and AFWAL/POSF. The required hardware included a mandrel fabricated by the Falex Corporation for use in mounting the Falex Ring to the standard BOCLE cylinder shaft, six Falex test rings (two each from three different lots), shims for BOCLE machine modification to enable leveling of the load arm and extension of hydraulic plunger, and 100 Falex test balls.

Two of the four fluid samples to be tested, Isopar M and an Isopar M + 30 ppm DCI-4A blend, were supplied by P&W. The remaining two test fluids, consisting of a petroleum based JP-4 and a shale derived CT JP-4, were supplied by AFWAL/POSF. The latter samples were test fluids which had been used in the December 1984 CRC BOCLE Task Force Round Robin. The samples had been in refrigerated storage since the completion of that program. The four test fuels were selected based on their range of wear scars, volatility extremes and availability of historical data generated using conventional BOCLE cylinders. All test fuels were evaluated in triplicate on each of the six Falex Rings.

5. TEST BALL INVESTIGATION

The final effort conducted under this task was an investigation of the effect of the test balls on BOCLE test results. This study focused on the dependence of wear scar size and repeatability on the source from which the ball was procured. A number of suppliers of 0.5 in., AISI 52100, Rc 64-66, Grade 25 EP ball bearings were contacted to determine the origin of the test balls traditionally used in conjunction with the BOCLE test apparatus.

SKF, a major distributor of ball bearings, advised that test balls acquired from SKF were manufactured both domestically and in Sweden. SKF manufactures their own ball bearings in Sweden. These are considered precision balls, and are produced under tight tolerances, with little or no variation from batch to batch. The balls are furnished with a Grade 5 to 10 EP polished finish, as opposed to the 25 EP standard equated with balls produced in the United States. Balls originating from SKF Sweden will always carry the designation "RB12.7" on the box in which they are packaged. The "12.7" part of the Swedish designation represents the diameter of the ball in millimeters.

Balls obtained domestically for distribution by SKF are procured from three different manufacturers and sold by SKF's Atlas Ball Division. Manufacturers which produce balls for Atlas include N.N. & Roller Co. of Erwin, New Jersey; Hoover Group, also of Erwin, New Jersey; and Winstead Precision Ball Co., located in Colebrook, Connecticut. Consequently, Atlas Balls would be expected to show slight batch to batch variations. The Falex Corporation, a major manufacturer of wear type equipment and supplier of 0.5 in. BOCLE balls, purchase their balls for resale from SKF's Atlas Ball Division. Another variety of ball, somewhat inaccurately referred to as "German Balls," although distributed in Germany, were found to be produced in SKF's plant in Sweden.

Test balls received from Falex, SKF Atlas, SKF Sweden, and Winstead were evaluated in BOCLE tests in four fuel types. The test fuels consisted of a petroleum based JP-4, JP-7, CT JP-4, and Isopar M + DCI-4A (BOCLE calibration/ reference fluid). Tests were performed in triplicate using the Falex Ring at a 500 g applied load.

SECTION III

RESULTS AND DISCUSSION

1. REFERENCE FLUID SELECTION

Preliminary screening of n-paraffins, cycloparaffins, and hydrocarbon mixtures resulted in the elimination of the C6 through C12 n-paraffins and cycloparaffins from further consideration. In these preliminary tests, abrasive wear (scuffing) was exhibited which in most cases resulted in premature termination of the test. Four of the more promising candidate fluids, which produced acceptable wear scars, were selected for in-depth evaluations. Those candidates shown to merit further investigation included neat Isopar M, an Isopar M + 30 ppm DCI-4A additive blend, tetralin, and a mixture of 15 volume percent (vol %) tetralin in Isopar M.

Table 1 shows BOCLE test results for the four candidate reference fluids (CRF). Cylinders identified by a "J" prefix were of known poor quality. Those labeled with a prefix of "FV" were previously qualified at 0.30 ± 0.02 mm WSD and were determined to be acceptable P&W control cylinders.

TABLE 1. (U)
BOCLE DATA FOR CANDIDATE REFERENCE FLUIDS

		Wear Scar Diameter, mm					
Candidate Fluid	*Run No.*	*Cyl. J14*	*Cyl. J01*	*Cyl. J27*	*Cyl. FV5*	*Cyl. FV6*	*Cyl. FV7*
Isopar M/DCI-4A	1	0.595	0.420	0.520	0.305	0.270	0.340
	2	0.590	0.465	0.535	0.305	0.290	0.310
	3	0.550	0.455	0.510	0.275	0.295	0.325
	4	0.530	0.430	0.505	0.320	0.235	0.320
Tetralin	1	0.395	0.320	0.365	0.285	0.280	0.295
	2	0.365	0.335	0.375	0.275	0.275	0.280
	3	0.375	0.505	0.365	0.260	0.285	0.290
	4	0.440	0.325	0.420	0.325	0.285	0.290
85 Vol % Isopar M/ 15 Vol % Tetralin	1	0.690	0.600	0.675	0.690	0.560	0.665
	2	0.580	0.565	0.675	0.645	0.595	0.660
	3	0.705	0.555	0.700	0.665	0.590	0.655
	4	0.650	0.540	0.635	0.650	0.640	0.645
Isopar M	1	> 1.00	0.920	1.02	0.945	0.930	0.945
	2	0.865*	1.01	0.805	0.925	0.895	0.905
	3	0.875	0.935	0.925	0.930	0.910	0.935
	4	0.950	0.920	0.965	0.895	0.940	0.910

* Scuffed

Note: Cylinders prefixed by 'J' are of known poor quality
Cylinders prefixed by 'FV' are of proven good quality

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a. Ability to Discern Cylinder Quality

As a method of assessing the data to determine which fluids were capable of differentiating between cylinders, the average WSD of the group of three good cylinders and the group of three bad cylinders were calculated for each fluid. These are shown in Table 2. As indicated, both tetralin and Isopar M + DCI-4A were able to differentiate between good cylinders and bad cylinders. Those fluids which were unable to discern between the cylinders, and as such, rated both groups similarly, included the 85/15 vol % Isopar M/tetralin blend and neat Isopar M. Similar results for individual cylinder ratings for the CRF are shown in Table 3. Table 3 summarizes the data generated during the BOCLE tests by providing the average and standard deviation of the four runs performed on each of the three good and three bad cylinders for each CRF.

TABLE 2. (U)
DIFFERENTIATION BETWEEN GROUPS OF CYLINDERS

	Bad Cylinders		*Good Cylinders*	
Candidate Fluid	x̄ WSD	sd	x̄ WSD	sd
Isopar M/DCI-4A	0.509	0.057	0.299	0.029
Tetralin	0.382	0.053	0.285	0.015
85/15 Vol % Isopar M/Tetralin	0.631	0.060	0.638	0.037
Isopar M	0.936	0.068	0.922	0.018

5038C

b. Reproducibility of Candidate Fluid

In addition to the average WSD and standard deviation calculated for the CRF for each of the good and bad cylinders, Table 3 also shows the maximum spread in WSD for each of the test fluids. In this manner, the reproducibility from cylinder to cylinder of the candidate fluids can be assessed for good cylinders. Data scatter is anticipated on those cylinders which are of poor quality and as such cannot be used to assess the reproducibility of the CRF.

Table 3 would indicate that tetralin and neat Isopar M, followed by Isopar M + DCI-4A, are the most reproducible CRF from cylinder to cylinder. Although neat Isopar M is shown to be a reproducible fluid by its minimal spread in WSD, the data shown in Table 2 suggest that it may not discriminate between cylinders of good and poor quality.

The most objective evaluation of repeatability must consider primarily the "good" cylinders. The bad cylinders would be expected to show scatter. However, if very little or no spread is observed in the case of the bad cylinders by a CRF, then it is possible that the CRF is not sensitive to surface irregularities and variations in hardness. If a large spread is noted in the bad cylinders by a CRF, then the assumption can be made that the CRF is sensitive to differences across the cylinder surface.

c. Repeatability Across Running Surface

A review of standard deviations reported in Table 3 indicates that repeatability is good for all CRF on each of the three good cylinders. With the exception of Isopar M + DCI-4A, the repeatability of the fluids evaluated on poor cylinders declined dramatically. Since the scatter did not occur on the good cylinders, it is apparently not due to increasing fluid harshness.

Isopar M + 30 ppm DCI-4A and technical grade tetralin were selected as the two most promising candidates for use as a standard BOCLE reference fluid. Selection was made on the basis of the best combination of cylinder differentiation, repeatability across the running surface, and reproducibility from cylinder to cylinder. Because tetralin is a known producer of peroxides, a short investigation was conducted to determine the effect of peroxides on lubricity.

5421C

TABLE 3. (U)
PRECISION OF CANDIDATE REFERENCE FLUIDS

		Bad Cylinders Wear Scar Diameter, mm			
Candidate Fluid		*J14*	*J01*	*J27*	*Max. △*
Isopar M/DCI-4A	$\bar{x}$ =	0.566	0.443	0.518	0.123
	sd =	0.031	0.021	0.013	
Tetralin	$\bar{x}$ =	0.394	0.371	0.381	0.023
	sd =	0.033	0.089	0.026	
85/15 Vol % Isopar M/Tetralin	$\bar{x}$ =	0.656	0.565	0.671	0.106
	sd =	0.056	0.025	0.027	
Isopar M	$\bar{x}$ =	0.922	0.946	0.924	0.024
	sd =	0.064	0.043	0.085	

		Good Cylinders Wear Scar Diameter, mm			
Candidate Fluid		*FV5*	*FV6*	*FV7*	*Max. △*
Isopar M/DCI-4A	$\bar{x}$ =	0.301	0.273	0.324	0.051
	sd =	0.019	0.027	0.012	
Tetralin	$\bar{x}$ =	0.286	0.281	0.289	0.008
	sd =	0.028	0.005	0.006	
85/15 Vol % Isopar M/Tetralin	$\bar{x}$ =	0.663	0.596	0.656	0.067
	sd =	0.020	0.023	0.008	
Isopar M	$\bar{x}$ =	0.924	0.919	0.923	0.005
	sd =	0.021	0.020	0.019	

* $\bar{x}$ and sd based on 4 runs

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Preliminary results showed that peroxides have a significant effect on fuel lubricity. A dramatic increase in WSD was observed when the peroxide content of a stressed reagent grade tetralin was lowered from 100 ppm to 40 ppm. Lowering the peroxide content from 40 ppm to less than 1 ppm, by percolating the tetralin through activated silica, had no significant affect on lubricity. Tetralin used during CRF testing was determined to contain 100 ppm peroxides. Typically, 40 to 50 ppm peroxide were measured from new, previously unopened, bottles and 100 ppm from opened bottles after minimal shelf storage. A BOCLE test was performed on tetralin containing peroxides below detectable levels to determine if peroxides were generated during testing. Samples taken after 15 minutes and after 30 minutes showed no peroxide formation.

Based on the poor storage stability and unpredictable effects of peroxide formation, tetralin was rejected as a CRF. Isopar M + 30 ppm DCI-4A was selected as the recommended standard reference fluid and was used throughout the remainder of the program effort.

2. TEST CYLINDER SURFACE FINISH INVESTIGATION

The test cylinder investigation was directed at defining material hardness and surface finish specifications for standardizing BOCLE test cylinders. The effect of cylinder surface finish on data precision is discussed in the following paragraphs.

After one-third of the planned BOCLE tests had been completed, a number of anomalies were noted. Testing was temporarily suspended and the data critically reviewed. As shown in

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Table 4, the average WSD for JP-7 closely approximated the WSD for the CT JP-4 sample on the J-Al cylinder. Typically, there is good differentiation between these two fuel types, JP-7 being considerably less harsh than a CT fuel. Since this was not reflected in the measurements, the data were considered suspect. Significantly higher WSDs than expected were exhibited by both the reference fluid (Isopar M + DCI-4A) and JP-4 on the 4 to 9 μin. J-Al cylinder. The nominal WSD indicative of these fluids is 0.30 to 0.33 mm . The WSD measured for the reference fluid was inconsistent with the WSD measured for JP-4 on the FV9 4 to 9 μin. cylinder in that the reference fluid was formulated to approximate a high lubricity JP-4. Conflicting WSD were reported for all but the CT JP-4 sample when comparing the two sources of 4 to 9 μin. cylinders. The source of the 4 to 9 μin. cylinders appeared to be contributing more to variations in reproducibility than the surface finish.

TABLE 4. (U)
EFFECT OF CYLINDER SURFACE FINISH — PART I

			Wear Scar Diameter, mm			
Cyl No.	*SF (μin.)*	*Run No.*	*ISOPAR M +DCI 4A*	*JP 4*	*JP 7*	*CT JP-4*
J-A1	4-9*	1	0.465	0.550	0.855	0.860
		2	0.460	0.560	0.840	0.895
		3	0.475	0.650	0.840	0.800
		4	0.475	0.530	0.845	0.970
		$\bar{x}$	0.469	0.572	0.845	0.881
		sd	0.008	0.053	0.007	0.071
		Δ	0.015	0.120	0.015	0.170
J-F1	20-30*	1	0.525	0.645	0.805	0.885
		2	0.505	0.570	0.780	0.765
		3	0.490	0.580	0.800	0.885
		4	0.485	0.630	0.795	0.895
		$\bar{x}$	0.501	0.606	0.795	0.858
		sd	0.018	0.037	0.011	0.062
		Δ	0.040	0.075	0.025	0.130
FV9	4-9	1	0.290	0.405	0.555	0.835
		2	0.305	0.445	0.610	0.860
		3	0.295	0.390	0.575	0.940
		4	0.275	0.380	0.575	0.900
		$\bar{x}$	0.291	0.405	0.579	0.884
		sd	0.012	0.028	0.023	0.046
		Δ	0.030	0.065	0.055	0.105

		All Fuels Repeatability (Avg. Spread)		*Cal Fluid*
		Δ	sd	$\bar{x}$
J-A1	4-9	0.080	0.035	0.469
J-F1	20-30	0.068	0.032	0.501
FV9	4-9	0.064	0.027	0.291

*Note: Post test Profilometer traces showed surface finish of cylinders fabricated by J inconsistent with specificiation.
J-A1 = 18.5 μin. J-F1 = 27 μin.

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The average data spread and the average standard deviation were calculated to assess the effect of both the source and the two different surface finishes on repeatability. This calculation is presented in Table 4 and is inclusive of all fuel types. This approach, in effect, gives insight into the average error that could be anticipated during a BOCLE test on any given fuel within the confines of those tested. The average potential error (Δ) is shown to be as great between the

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two different sources of the same surface finish, as it is between the two distinctive surface finishes evaluated. This is also true of the average WSD shown for the calibration fluid for each of the three cylinders. The difference in the average WSD for the calibration fluid is as great between the two different 4 to 9 µin. cylinder sources as it is between the 4 to 9 µin. and 20 to 30 µin. finishes.

Surface finish and hardness measurements were performed on the above cylinders to verify that they had been fabricated to specification by the two different vendors. While cylinder hardnesses proved to be within specification limits, profilometer measurements indicated that the majority of the J manufactured cylinders failed to meet specification for surface finish. As shown in Table 5, only four were found to be within the specification limits. Surface finish varied from 11 to 40 µin. on the remaining J cylinders fabricated to a 4 to 9 µin. specification. The surface finishes of the FV cylinders, fabricated and ground to a 4 to 9 µin. specification were also verified by profilometer measurements. All FV cylinders were found to be within specification limits.

TABLE 5.
VERIFICATION OF CYLINDER SURFACE FINISH

Source:	*FV*	*J*	
Purchased Specification:	*4-9 µin.*	*4-9 µin.*	*20-30 µin.*
	(Number of Cylinders)		
Surfanalyzer Ra* Results, µin.			
1-10	10	0	
11-20		14	2
21-30		9	8
30-40		2	5

*Ra — Roughness Average
Average of 2 Ra values taken at 90 degrees

The results of the profilometer measurements for each set of cylinders shown in Table 5 are based on an average of two readings across the cylinder surface. Two locations were measured, the second at a 90 degree rotation from the first. Twelve of the 40 J cylinders had a difference in Ra (roughness average) value of greater than 5 µin. This indicates a significant difference in the surface finish around the cylinder. In contrast, the FV cylinders had a maximum difference of 2 µin. (1 of the 10 cylinders), with 7 of the 10 cylinders having a difference of 1 µin.

Based on these findings, the previously performed tests were considered invalid for the purpose of evaluating surface finish. They did demonstrate, however, the importance of vendor quality control. Testing was resumed and was restricted to assessing the effects of two different verified surface finishes on test repeatability. The second set of BOCLE tests which followed compared three FV fabricated cylinders with verified surface finishes of 4 to 9 µin. to that of three J fabricated cylinders with verified 16 to 22 µin. surface finishes. The same test fluids were used as in the previous analyses. The results are shown in Table 6.

TABLE 6.
EFFECT OF CYLINDER SURFACE FINISH — PART II

			Wear Scar Diameter, mm			
Cyl No.	*SF (μin.)*	*Run No.*	*ISOPAR M +DCI 4A*	*JP 4*	*JP 7*	*CT JP-4*
FV22	4-9	1	0.235	0.470	0.530	1.03
		2	0.245	0.420	0.600	0.840
		3	0.275	0.475	0.595	0.960
		4	0.270	0.460	0.575	0.935
		$\bar{x}$	0.256	0.456	0.575	0.941
		st	0.098	0.025	0.032	0.078
		△	0.040	0.055	0.070	0.190
FV23	4-9	1	0.285	0.610	0.835	0.905
		2	0.300	0.470	0.675	1.010
		3	0.340	0.445	0.590	0.980
		4	0.315	0.510	0.675	0.945
		$\bar{x}$	0.310	0.509	0.694	0.960
		st	0.023	0.073	0.102	0.045
		△	0.055	0.165	0.245	0.105
FV25	4-9	1	0.335	0.525	0.825	1.050
		2	0.315	0.490	0.740	0.830
		3	0.325	0.495	0.750	0.995
		4	0.265	0.505	0.760	0.940
		$\bar{x}$	0.310	0.502	0.769	0.954
		sd	0.031	0.016	0.038	0.094
		△	0.070	0.035	0.085	0.220
J-F2	16-22	1	0.600	0.755	0.805	0.970
		2	0.560	0.635	0.800	0.880
		3	0.525	0.645	0.775	0.865
		4	0.445	0.680	0.785	0.950
		$\bar{x}$	0.532	0.679	0.791	0.916
		st	0.066	0.054	0.014	0.052
		△	0.155	0.120	0.030	0.105
J-F3	16-22	1	0.550	0.690	0.795	0.975
		2	0.530	0.635	0.820	0.935
		3	0.555	0.565	0.815	0.900
		4	0.510	0.570	0.840	0.930
		$\bar{x}$	0.536	0.615	0.818	0.935
		st	0.021	0.059	0.018	0.031
		△	0.045	0.120	0.045	0.075
J-F5	16-22	1	0.510	0.600	0.765	0.930
		2	0.480	0.595	0.750	0.940
		3	0.465	0.585	0.800	0.990
		4	0.465	0.560	0.815	0.975
		$\bar{x}$	0.480	0.585	0.782	0.959
		sd	0.021	0.018	0.030	0.028
		△	0.045	0.040	0.065	0.060

		Repeatability (avg. spread)		*Reproducibility between 3 cylinders (Max. △between avg. cylinder value)*				
		△ *All Fuels*	*sd*	*Cal Fl*	*JP 4*	*JP 7*	*CT JP 4*	$\bar{x}$ Max △
FV	4-9	0.111	0.048	0.054	0.053	0.194	0.013	0.078
J	16-22	0.075	0.034	0.056	0.094	0.036	0.043	0.057

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To summarize and provide an overview of the test data, such that a quick comparison of the two surface finishes could be made, average spread and maximum differences between average cylinder WSD were calculated for each fuel type. Average spread was determined by averaging the deltas (highest WSD - lowest WSD) from each of the four BOCLE runs performed on each fuel for each of the three 4 to 9 μin. cylinders. This average spread was then compared to that calculated for the 16 to 22 μin. cylinders. This summary is shown at the bottom of Table 6. Average spread was used as a means of assessing repeatability across the running surface of the test cylinders. The maximum difference between the average cylinder WSD was used to evaluate reproducibility between cylinders of the same surface finish. Although the test results indicated that the 4 to 9 μin. FV cylinders were somewhat superior to that of the 16 to 22 μin. J cylinders, the desired precision was not achieved by either surface finish. The overall repeatability of this series of tests was, in fact, worse than the preceding series of tests whereby the surface finish specifications had not been met. The data was, therefore, once more considered inconclusive.

A tighter controlled test plan was devised in an effort to eliminate any unknown variables which may have contributed to the data scatter encountered in the former series of BOCLE tests. In the subsequent third set of tests, two FV 4 to 9 μin. cylinders were compared to two J 16 to 22 μin. cylinders. In contrast to the former surface finish evaluations, the same operator performed all test runs. Periods of interruption in the completion of the test matrix were minimized. All runs were restricted to a single BOCLE unit, whereas previous testing had been conducted simultaneously on two separate InterAv BOCLE units. Spacing of run tracks was also a concern. The previous series of tests were characterized by relatively close spacing (0.5 mm) between run tracks on the test cylinder. There was some concern that localized heating and surface deformation caused by a previous run may have some effect on a succeeding run. The follow-up series of tests used 0.75 mm spacing between tracks. These tests were performed in triplicate using three of the four original test fluids. The results of this third series of tests are shown in Table 7.

Repeatability was again related to the average spread observed inclusive of all fuels. In a similar manner, reproducibility from cylinder to cylinder was related to the maximum difference calculated between average cylinder WSD. Table 7 shows that there was some improvement in repeatability of the 4 to 9 μin. cylinders based on the average spread. Reproducibility also showed improvement. However, in this case only two cylinders of each surface finish were evaluated which inherently biases comparison. The conclusions afforded by the test results were disappointing in that the desired repeatability required for selection of an optimum surface finish still had not been achieved.

3. FALEX RING INVESTIGATION

A comprehensive investigation of the Falex Ring resulted from the inability to identify an optimum surface finish or a source for reliable repeatable conventional BOCLE test cylinders. The results of this investigation are discussed in the following paragraphs.

Table 8 shows data generated in a preliminary investigation of the Falex Ring. The purpose of this preliminary investigation was to determine the merit of conducting a full-scale evaluation of the Falex Ring as a potential candidate for replacing the conventional test cylinder. As shown in Table 8, the test data were very promising. Repeatability across the running surface was excellent. Little variation from ring to ring was observed for the three rings tested. The desired differentiation between fuel types was apparent and the size of the wear scar exhibited little influence on data scatter.

TABLE 7.
EFFECT OF CYLINDER SURFACE FINISH — PART III

Cyl No.	SF (μin.)	Run No.	Wear Scar Diameter, mm: Isopar M DCI-4A	JP-4	JP-7
FV25	4-9	1	0.315	0.495	0.595
		2	0.355	0.470	0.670
		3	0.320	0.460	0.665
		$\bar{x}$	0.330	0.475	0.643
		st	0.022	0.018	0.042
		△	0.040	0.035	0.075
FV13	4-9	1	0.330	0.570	0.720
		2	0.295	0.460	0.730
		3	0.310	0.410	0.620
		$\bar{x}$	0.312	0.480	0.690
		st	0.018	0.082	0.061
		△	0.035	0.160	0.110
J-F2	16-22	1	0.460	0.625	0.800
		2	0.470	0.680	0.785
		3	0.455	0.670	0.760
		$\bar{x}$	0.462	0.658	0.782
		sd	0.008	0.029	0.020
		△	0.015	0.055	0.040
J-F3	16-22	1	0.440	0.505	0.785
		2	0.465	0.855	0.775
		3	0.450	0.485	0.820
		$\bar{x}$	0.452	0.615	0.793
		st	0.013	0.208	0.024
		△	0.025	0.370	0.045

		Repeatability (avg. spread) △ All Fuels	sd	Reproducibility between 2 cylinders (Max. △between avg. cylinder value) Cal Fluid	JP-4	JP-7
FV	4-9	0.078	0.041	0.018	0.005	0.047
J	16-22	0.092	0.050	0.010	0.043	0.011

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The wear scar generated on the Falex Ring was found to be well-defined. Measurement of the wear scar was considerably less subjective than that of the conventional BOCLE cylinder. Figure 2 illustrates the irregular and jagged wear scars produced by the conventional cylinders for three typical jet fuels. Unlike these ill-defined scars, the Falex Ring scars were well-defined, symmetrical ellipses.

Harsh fluids, which in the past were found to be nonreproducible as a result of the scatter induced by large wear scars, were found to show excellent repeatability. Based on these preliminary results, it was concluded that the Falex Ring appeared to enhance test precision and merited a full-scale investigation.

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TABLE 8.
PRELIMINARY FALEX RING EVALUATION

		Wear Scar Diameter, mm				
Ring No.	*Run No.*	*JP-7 Tank 2*	*Isopar M DCI-4A*	*JP-4 Tank 1*	*CT JP-4*	*JP-8 WPAFB*
3	1	0.680	0.520	0.545	0.935	0.550
	2	0.680	0.515	0.550	0.905	0.560
	3	0.685	0.520	0.545	0.880	0.540
	$\bar{x}$	0.682	0.518	0.547	0.907	0.550
	sd	0.003	0.003	0.003	0.028	0.010
	△	0.005	0.005	0.005	0.055	0.020
4	1	0.680	0.520	0.565	0.905	0.560
	2	0.685	0.530	0.555	0.915	0.555
	3	0.685	0.505	0.550	0.895	0.555
	$\bar{x}$	0.683	0.518	0.557	0.905	0.557
	sd	0.003	0.013	0.008	0.010	0.003
	△	0.005	0.025	0.015	0.020	0.005
5	1	0.680	0.515	0.565	0.935	0.560
	2	0.675	0.510	0.565	0.945	0.555
	3	0.680	0.510	0.550	0.930	0.545
	$\bar{x}$	0.678	0.512	0.560	0.937	0.553
	sd	0.003	0.003	0.009	0.008	0.008
	△	0.005	0.005	0.015	0.015	0.015

Repeatability (avg. spread)		*Reproducibility between 3 rings (Max. △between avg. ring value)*				
△ *All Fuels*	*sd*	*JP-7*	*Cal Fluid*	*JP-4*	*CT JP-4*	*JP-8*
0.014	0.009	0.005	0.006	0.013	0.032	0.007

Applied Load: 500 g

Falex Ring Specification:
Material: SAE 8720
Hardness: 58-62 Rc
Surface Finish: 20-30 μin

Table 9 lists the data from BOCLE tests performed on four different production lots of Falex Rings. The data summarized at the bottom of Table 9 present an assessment of the Falex Ring in terms of both repeatability and reproducibility between material lots. The data generated exhibited excellent repeatability across the running surface of any given test ring, within any given lot, for all fuel types tested. The loss of repeatability and introduction of data scatter with increasing fuel harshness, as typically experienced with conventional BOCLE cylinders, was not exhibited by the Falex Ring.

The Falex Ring also exhibited excellent [illegible] reproducibility. Figure 3 illustrates the minimal variation in Falex lots. In this plot, the average WSD for each lot is shown as a function of fuel type. The average WSD for the four lots differed by a maximum of only 0.006 to 0.029 mm for the four high-to-intermediate lubricity level fluids. The maximum variation between averages of the four lots was only 0.028 mm for the harshest of the test fluids (CT JP-4). Small standard deviations, calculated for all runs performed on each test fluid, indicate a very close grouping about the means.

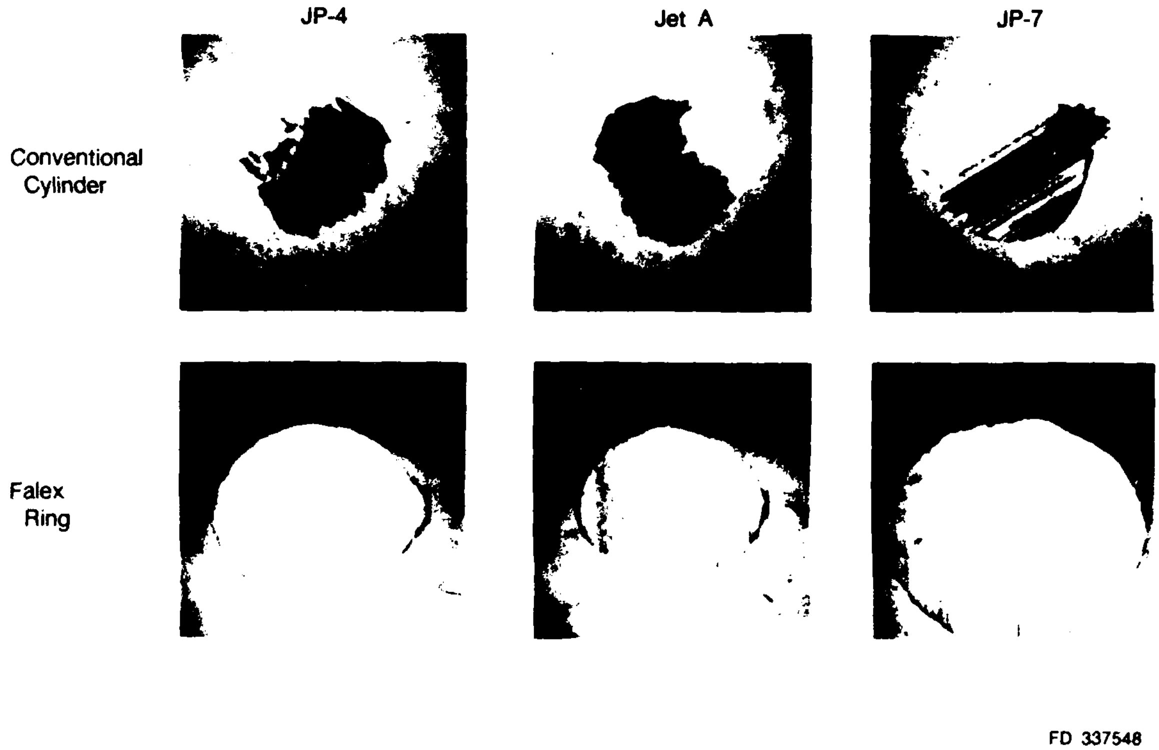

Figure 2. — Wear Scar Interpretation

Based on the average WSD for the 36 runs made on each of the test fluids, the fluids shown in Table 9 were grouped in order of descending lubricity levels. The relative ranking of the fluids in terms of decreasing fuel lubricity (increasing WSD) were as follows: Isopar M + 30 ppm DCI-4A (0.504) > JP-4 (0.552) / JP-8 (0.556) > JP-7 (0.684) > CT JP-4 (0.917). Discrimination between JP-4 and JP-8 was not obvious. WSD differed by only 0.006 mm, which is well within the repeatability of the test. There is no data bank currently available which characterizes the lubricity properties of JP-8. However, the data bank generated for the remaining test fluids indicates that the Falex Ring provides good differentiation between fuel types of known lubricity levels.

As a cursory check on quality control, six Falex Rings were analyzed to confirm that specification limits had been met during production. Surface finishes and hardnesses all fell within a tight tolerance band conforming to specification.

4. INTERLABORATORY EVALUATION OF THE FALEX RING

Results from the five laboratories completing the Falex Ring Round Robin are shown in Tables 10 through 13. One laboratory, due to a shortage in available manpower, was able to evaluate only one ring per material lot for each of the three different lots. The remaining laboratories evaluated two rings from each lot. The mean, standard deviation, and range are shown, as a function of fuel type, for each laboratory. This serves to provide a cursory assessment of repeatability and reproducibility among laboratories.

TABLE 9.
REPRODUCIBILITY BETWEEN FALEX LOTS

		Wear Scar Diameter, mm				
Ring No.	*Run No.*	*Isopar M +DCI-4A*	*JP-4*	*JP-8*	*JP-7*	*CT JP-4*
K-1	1	0.490	0.525	0.535	0.670	0.885
	2	0.475	0.525	0.535	0.665	0.905
	3	0.470	0.530	0.540	0.675	0.910
	x	0.478	0.527	0.537	0.670	0.900
	sd	0.010	0.003	0.003	0.005	0.013
	max.Δ	0.020	0.005	0.005	0.010	0.025
K-2	1	0.500	0.540	0.555	0.675	0.890
	2	0.505	0.550	0.555	0.695	0.895
	3	0.510	0.555	0.550	0.695	0.930
	x	0.505	0.548	0.553	0.688	0.905
	sd	0.005	0.008	0.003	0.012	0.022
	max.Δ	0.010	0.015	0.005	0.020	0.040
K-3	1	0.485	0.565	0.545	0.680	0.910
	2	0.510	0.570	0.575	0.695	0.920
	3	0.515	0.565	0.570	0.695	0.900
	x	0.503	0.567	0.563	0.690	0.910
	sd	0.016	0.003	0.016	0.009	0.010
	max.Δ	0.030	0.005	0.030	0.015	0.020
L-1	1	0.510	0.550	0.555	0.670	0.895
	2	0.485	0.550	0.545	0.675	0.910
	3	0.505	0.555	0.565	0.665	0.920
	x	0.500	0.552	0.555	0.670	0.908
	sd	0.013	0.003	0.010	0.005	0.013
	max.Δ	0.025	0.005	0.020	0.010	0.025
L-2	1	0.500	0.540	0.555	0.680	0.900
	2	0.500	0.560	0.565	0.700	0.900
	3	0.490	0.560	0.555	0.690	0.930
	x	0.497	0.553	0.558	0.690	0.910
	sd	0.006	0.012	0.006	0.010	0.017
	max.Δ	0.010	0.020	0.010	0.020	0.030
L-3	1	0.500	0.540	0.565	0.685	0.925
	2	0.495	0.550	0.565	0.710	0.910
	3	0.505	0.550	0.565	0.690	0.930
	x	0.500	0.547	0.565	0.695	0.922
	sd	0.005	0.006	0.000	0.013	0.010
	max.Δ	0.010	0.010	0.000	0.025	0.020
M-1	1	0.525	0.550	0.545	0.680	0.915
	2	0.480	0.555	0.580	0.680	0.905
	3	0.505	0.545	0.550	0.670	0.905
	x	0.503	0.550	0.558	0.677	0.908
	sd	0.023	0.005	0.019	0.006	0.006
	max.Δ	0.045	0.010	0.035	0.010	0.010
M-2	1	0.500	0.555	0.550	0.675	0.925
	2	0.530	0.580	0.555	0.690	0.950
	3	0.520	0.565	0.560	0.690	0.935
	x	0.517	0.567	0.555	0.685	0.937
	sd	0.015	0.013	0.005	0.009	0.013
	max.Δ	0.030	0.025	0.010	0.015	0.025

TABLE 9.
REPRODUCIBILITY BETWEEN FALEX LOTS (CONTINUED)

		Wear Scar Diameter, mm				
Ring No.	*Run No.*	*Isopar M +DCI-4A*	*JP-4*	*JP-8*	*JP-7*	*CT JP-4*
M-3	1	0.500	0.555	0.580	0.695	0.945
	2	0.490	0.545	0.550	0.695	0.980
	3	0.505	0.555	0.565	0.705	0.935
	x	0.498	0.552	0.565	0.698	0.953
	sd	0.008	0.006	0.015	0.006	0.024
	max.△	0.015	0.010	0.030	0.010	0.045
MX-3	1	0.520	0.545	0.550	0.680	0.935
	2	0.515	0.550	0.560	0.680	0.905
	3	0.520	0.545	0.540	0.685	0.880
	x	0.518	0.547	0.550	0.682	0.907
	sd	0.003	0.003	0.010	0.003	0.028
	max.△	0.005	0.005	0.020	0.005	0.055
MX-4	1	0.520	0.565	0.560	0.680	0.905
	2	0.530	0.555	0.555	0.685	0.915
	3	0.505	0.550	0.555	0.685	0.895
	$\bar{x}$	0.518	0.557	0.557	0.683	0.905
	sd	0.013	0.008	0.003	0.003	0.010
	max.△	0.025	0.015	0.005	0.005	0.020
MX-5	1	0.515	0.565	0.560	0.680	0.935
	2	0.510	0.565	0.555	0.675	0.945
	3	0.510	0.550	0.545	0.680	0.930
	x	0.512	0.560	0.553	0.678	0.937
	sd	0.003	0.009	0.008	0.003	0.008
	max.△	0.005	0.015	0.015	0.005	0.015
Lot avg.						
K		0.496	0.547	0.551	0.683	0.905
L		0.499	0.551	0.559	0.685	0.913
M		0.506	0.556	0.559	0.687	0.933
MX		0.516	0.553	0.553	0.681	0.916
All runs	$\bar{x}$	0.504	0.552	0.556	0.684	0.917
All runs	sd	0.015	0.012	0.011	0.011	0.021
Lot-Lot	max.△*	0.020	0.009	0.008	0.006	0.028

*Calculated on average value for each lot.

Summary of Test Matrix

No. of lots evaluated: 4
No. of rings/lot: 3
No. of fuel samples: 5
No. of runs/fuel sample: 36
No. of total runs: 180

Special Test Conditions

Applied load: 500 g

Falex Ring Specification

Material: SAE 8720
Hardness: 58-62 Rc
Surface finish: 20-30 μin.

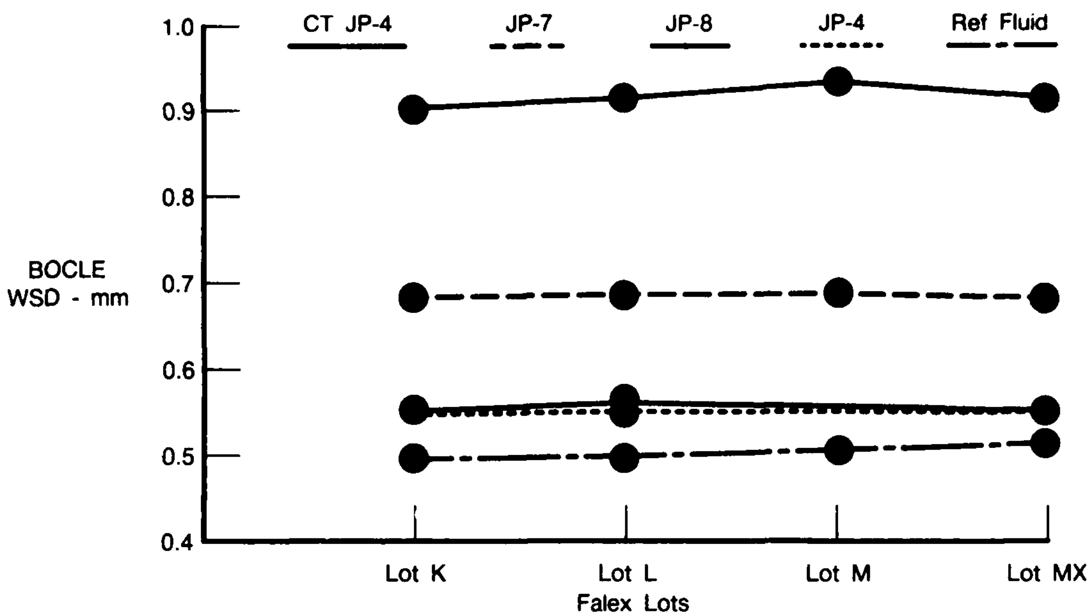

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Figure 3. — Variation in Falex Lots

TABLE 10.
FALEX RING ROUND ROBIN RESULTS FOR ISOPAR M +30 PPM DCI-4A

Lot Desig.	*Ring No.*	*Run No.*	*Pratt & Whitney*	*Woodward Governor*	*Chevron*	*Rolls Royce*	*WPAFB*
K	1	1	0.510	0.510	0.510	0.496	0.500
		2	0.485	0.520	0.510	0.510	0.505
		3	0.485	0.520	0.530	0.507	0.505
	2	1	0.490	0.520		0.504	0.500
		2	0.495	0.530		0.481	0.510
		3	0.490	0.530		0.511	0.520
KX	1	1	0.500	0.530	0.510	0.499	0.510
		2	0.510	0.540	0.500	0.506	0.530
		3	0.520	0.520	0.520	0.488	0.510
	2	1	0.490	0.510		0.479	0.525
		2	0.505	0.510		0.501	0.490
		3	0.515	0.520		0.473	0.515
L	1	1	0.475	0.530	0.510	0.476	0.530
		2	0.485	0.530	0.520	0.500	0.510
		3	0.485	0.540	0.540	0.515	0.525
	2	1	0.520	0.530		0.477	0.515
		2	0.525	0.540		0.521	0.490
		3	0.515	0.520		0.501	0.525
Mean			0.500	0.525	0.517	0.497	0.512
Standard Deviation			0.015	0.010	0.012	0.015	0.012
Range			0.050	0.030	0.040	0.048	0.040

TABLE 11.
FALEX RING ROUND ROBIN RESULTS FOR JP-4

Lot Desig.	*Ring No.*	*Run No.*	*Pratt & Whitney*	*Woodward Governor*	*Chevron*	*Rolls Royce*	*WPAFB*
K	1	1	0.565	0.570	0.590	0.586	0.555
		2	0.565	0.570	0.580	0.595	0.560
		3	0.565	0.580	0.570	0.597	0.555
	2	1	0.560	0.570		0.564	0.560
		2	0.575	0.570		0.568	0.570
		3	0.570	0.570		0.585	0.575
KX	1	1	0.555	0.600	0.580	0.616	0.575
		2	0.565	0.580	0.570	0.553	0.565
		3	0.555	0.600	0.590	0.593	0.560
	2	1	0.575	0.590		0.580	0.540
		2	0.560	0.590		0.570	0.565
		3	0.560	0.590		0.588	0.555
L	1	1	0.545	0.590	0.590	0.582	0.585
		2	0.555	0.580	0.580	0.577	0.565
		3	0.565	0.570	0.580	0.561	0.565
	2	1	0.570	0.570		0.584	0.570
		2	0.570	0.580		0.567	0.555
		3	0.575	0.580		0.581	0.575
Mean			0.564	0.581	0.581	0.580	0.564
Standard Deviation			0.008	0.011	0.008	0.015	0.010
Range			0.030	0.030	0.020	0.063	0.045

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TABLE 12.
FALEX RING ROUND ROBIN RESULTS FOR ISOPAR M

Lot Desig.	*Ring No.*	*Run No.*	*Pratt & Whitney*	*Woodward Governor*	*Chevron*	*Rolls Royce*	*WPAFB*
K	1	1	0.840	0.840	0.820	0.804	0.790
		2	0.820	0.860	0.800	0.824	0.805
		3	0.845	0.850	0.820	0.808	0.820
	2	1	0.825	0.830		0.827	0.790
		2	0.835	0.840		0.823	0.825
		3	0.830	0.830		0.814	0.795
KX	1	1	0.860	0.840	0.800	0.862	0.820
		2	0.840	0.860	0.800	0.803	0.810
		3	0.845	0.860	0.810	0.855	0.790
	2	1	0.850	0.840		0.829	0.795
		2	0.845	0.840		0.800	0.810
		3	0.840	0.840		0.833	0.830
L	1	1	0.840	0.840	0.830	0.826	0.815
		2	0.855	0.850	0.820	0.812	0.790
		3	0.855	0.850	0.820	0.824	0.795
	2	1	0.810	0.850		0.812	0.800
		2	0.810	0.850		0.817	0.820
		3	0.830	0.860		0.820	0.790
Mean			0.838	0.846	0.813	0.822	0.805
Standard Deviation			0.014	0.010	0.011	0.016	0.014
Range			0.050	0.030	0.030	0.062	0.040

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TABLE 13.
FALEX RING ROUND ROBIN RESULTS FOR CLAY TREATED SHALE JP-4

Lot Desig.	*Ring No.*	*Run No.*	*Pratt & Whitney*	*Woodward Governor*	*Chevron*	*Rolls Royce*	*WPAFB*
K	1	1	0.855	0.900	0.930	0.777	0.800
		2	0.855	0.910	0.900	0.815	0.795
		3	0.835	0.920	0.890	0.847	0.785
	2	1	0.865	0.930		0.844	0.740
		2	0.835	0.910		0.823	0.790
		3	0.825	0.930		0.773	0.775
KX	1	1	0.860	0.920		0.844	0.775
		2	0.830	0.890	0.880	0.815	0.765
		3	0.825	0.920	0.880	0.851	0.760
	2	1	0.870	0.930		0.833	0.740
		2	0.850	0.920		0.854	0.735
		3	0.845	0.910		0.839	0.745
L	1	1	0.855	0.920	0.930	0.819	0.730
		2	0.845	0.910	0.860	0.810	0.715
		3	0.845	0.920	0.940	0.840	0.710
	2	1	0.820	0.900		0.822	0.760
		2	0.830	0.910		0.812	0.760
		3	0.830	0.910		0.856	0.750
Mean			0.843	0.914	0.901	0.826	0.757
Standard Deviation			0.015	0.011	0.029	0.024	0.026
Range			0.050	0.040	0.080	0.083	0.090

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A statistical analysis was performed on the raw test data using a Statistical Analysis System (SAS) computer software package and the general linear models procedure. The procedure used in the statistical analysis was as outlined in the "Manual On Determining Precision Data for ASTM Methods on Petroleum Products and Lubricants (RR-D-2-1007)." Statistically, a model was designed to assess the effect of the Falex Ring on test method precision. The model considered all effects (independent variables) encountered in the performance of the interlaboratory round robin. Construction of the model considered primary effects (labs, fuels, lots, rings) and indirect effects or interactions (lab*fuel, lab*lot, etc.).

Therefore,

Falex Ring Lubricity Model = Primary Effects + Interactions

= Labs + Lots + (Lab*Lot) + Rings + (Lab*Ring) + Fuels + (Lab*Fuel)

+ (Lot*Fuel) + (Ring*Fuel) + (Lab*Lot*Fuel) + (Lab*Ring*Fuel)

The general linear models procedure was used to determine deviations from the model. This process is based on laboratory observations and model-predicted values. From these deviations, differences in the levels of the effects (both primary and interactions) were analyzed. Null and alternative hypotheses were formulated to evaluate the performance of the Falex Ring. Probability testing involved comparison by SAS of a calculated F value to that of a critical F value. If the probability of observing the calculated F value was small, or F itself was large, then the null hypothesis was rejected. It was, therefore, concluded that a statistically significant difference existed to support the alternative hypothesis. Conclusions, based on statistical analysis of the raw data, are discussed in the following paragraphs.

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There appeared to be no significant difference between Falex Ring material lots, nor any interactions between lot and other independent variables. Since all lot effects and interactions appeared to have no significant contribution to the model, all lots were considered identical, and the lot effect and lot interaction terms were dropped from the model. The model, consequently, was reduced to:

Falex Ring Lubricity Model = Labs + Rings + (Lab*Ring) + Fuels + (Lab*Fuel)

+ (Ring*Fuel) + (Lab*Ring*Fuel)

From the reduced model, an analysis of variance using the general linear models procedure in SAS was performed. Based on this test, it was concluded that: (1) there was a statistically significant difference in the four fuels as measured by the Falex Ring, (2) there was a significant difference between at least two laboratories measuring the CT JP-4 sample, and (3) there was no apparent difference in rings from ring to ring.

The aforementioned statistical tests were directed at the primary effects influencing the Falex Ring Lubricity Model. Interaction terms were evaluated similarly and were concluded to be insignificant. Hence, secondary effects involving interactions between rings and fuels and between rings and laboratories were also eliminated from the equation. Since there was shown to be no significant difference in rings, the ring term was eliminated from the Falex model along with the ring interaction terms. As a consequence, the model was reduced to:

Falex Ring Lubricity Model = Labs + Fuels + (Lab*Fuel)

Examination of data dispersion showed that CT JP-4 occurred at three distinct levels, as opposed to one unique level. As a result of this inconsistency, the entire statistical analysis was repeated considering only the three remaining fuel types. The F values for tests which yielded significant F values previously, were still significant after removal of the CT JP-4 data. Therefore, after elimination of the CT JP-4 data, the previous conclusions were unchanged.

A further analysis was performed to provide additional information on the effect of fuel type on reproducibility of data between laboratories. In this analysis, the mean WSD was determined for all runs as a function of fuel type, rings, and lots for each laboratory. The mean WSD as shown in Tables 10 through 13 for each laboratory were used in calculating the interlaboratory range for specific fuel types. This range was used to determine if there was an interaction between laboratory and fuel type. It was already determined statistically that an interaction between laboratory and fuel type was apparent with the CT JP-4 sample. This was supported by concern voiced by the supplier of the CT sample during the course of the round robin. Because the five CT samples were drawn from the bottom of a 55 gallon drum on different days over a period of 4 to 6 weeks, there was considerable doubt that the samples had identical compositions. Based on the range of WSD exhibited by the remaining fuels, little interaction between fuel type and laboratory were noted. Differences in average values for fuel types between all laboratories were as follows: Isopar M + DCI-4A, 0.028; JP-4, 0.017; neat Isopar M, 0.041; and CT JP-4, 0.157.

Elimination of the CT sample data, and a determination that an interlaboratory range of up to 0.041 was acceptable, would further reduce the practical model to:

Falex Ring Lubricity Model = Fuel

The statistical model, therefore, indicates that in theory, the Falex Ring measurement is a direct function of fuel lubricity.

A precision statement was calculated using the equations set forth for repeatability and reproducibility in "Manual On Determining Precision Data For ASTM Methods On Petroleum Products and Lubricants (RR D-2-1007)." The procedure consists of estimating variance components from a two-way analysis of variance. It is performed with regard for substitutions made for missing or outlying values. These components are then combined to provide estimates of repeatability and reproducibility variances. These terms provide a measure from which no two successive data points should differ within a laboratory, in the case of repeatability, or between laboratories when referencing reproducibility.

For lubricity measurements inclusive of all fuel types:

Repeatability, r = 0.042

Reproducibility, R = 0.262

For lubricity measurements excluding the CT JP-4 sample:

Repeatability, r = 0.035

Reproducibility, R = 0.108

5. FALEX RING LUBRICITY VALUES FOR TYPICAL JET FUELS

Commencing with the preliminary investigation and maintained throughout the remainder of this test program, all incoming fuel samples to the P&W facilities were monitored using the Falex Ring. Fuel samples were obtained from on-going engine tests and weekly sampling of on-site fuel storage tanks, and from a number of fuels of varying origin and types from foreign and domestic air bases. These data were used to initiate a data base for establishing Falex Ring lubricity values for typical jet fuels. This limited data bank also encompasses the data generated during the mini-round robin and is considered representative of expected values. Nominal WSD and those WSD which are speculated to be marginal for typical jet fuels are shown in Table 14 and Figure 4.

TABLE 14.
FALEX RING LUBRICITY VALUES FOR TYPICAL JET FUELS

Fuel Type	*Nominal WSD, mm*	*Marginal WSD, mm*
JP-4	0.550	0.600
JP-5	0.510	0.600
JP-7	0.690	0.740
Jet A	0.510	0.600
*JP-8	0.560	0.600
CT JP-4	0.920	

Calibrating Fluid: Isopar M +30 ppm DCI-4A
Falex Ring Deemed Acceptable With a Generated WSD of 0.500 ±0.020 mm.

*JP-8 Nominal WSD values based on limited data

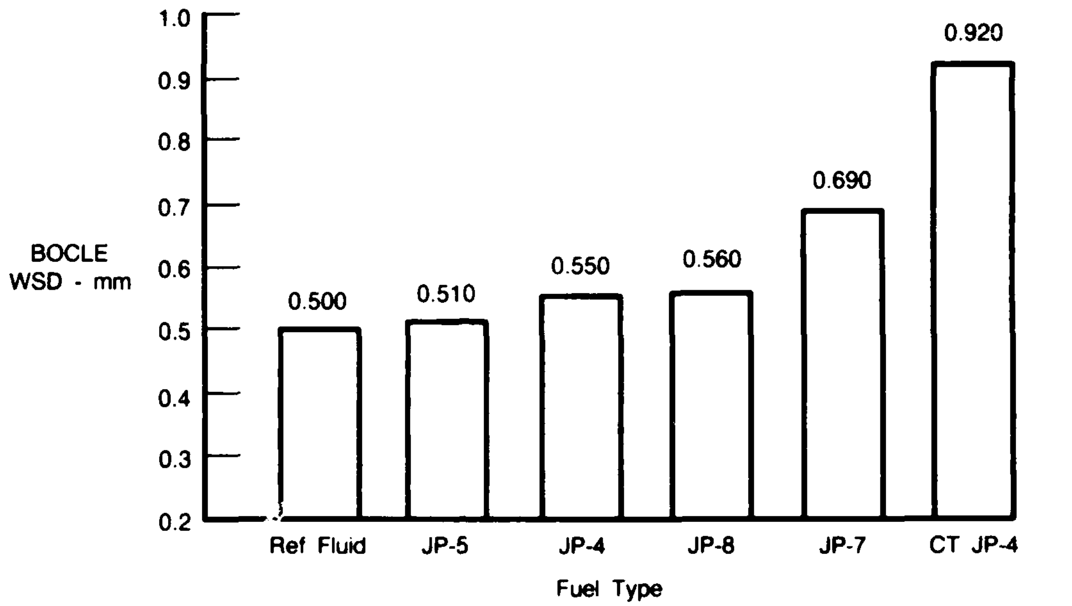

Figure 4. — Nominal Falex Ring Lubricity Values for Typical Jet Fuels

6. TEST BALL INVESTIGATION

The data in Table 15 show the influence that the source of the test ball can have on the size and repeatability of the BOCLE wear scar. BOCLE tests indicated that: (1) the Winstead ball was unable to differentiate between JP-4 and JP-7, and only marginally between JP-7 and the Isopar M / DCI-4A reference fluid; (2) the Winstead ball consistently produced lower WSD for all fluids; (3) the Falex, Atlas, and Swedish balls produced essentially the same average WSD for each specific fuel type; and (4) the ball source had no measurable effect on repeatability. Table 16 compares the composition of the SKF Swedish and SKF domestically produced 1/2 in. test balls.

The test results show that the source of the test ball can have a significant effect on the data generated during BOCLE operation. Material lots can vary as a consequence of the three different suppliers of balls to the Atlas Division of SKF. The balls produced by Winstead Precision Ball Co., one of the three potential Atlas suppliers, were shown to produce WSD inconsistent with those of the other two SKF suppliers.

TABLE 15.
TEST BALL SOURCE EVALUATION

		Wear Scar Diameter, mm			
Fuel Type	*Run No.*	*Falex*	*Atlas*	*Swedish*	*Winstead*
JP-4	1	0.560	0.560	0.560	0.445
	2	0.565	0.545	0.550	0.435
	3	0.560	0.570	0.535	0.460
	$\bar{x}$	0.562	0.558	0.548	0.447
	sd	0.003	0.013	0.013	0.013
JP-7	1	0.690	0.675	0.685	0.440
	2	0.695	0.690	0.675	0.460
	3	0.695	0.690	0.690	0.450
	$\bar{x}$	0.693	0.685	0.683	0.450
	sd	0.003	0.009	0.008	0.010
CT JP-4	1	0.905	0.900	0.925	0.700
	2	0.950	0.925	0.915	0.725
	3	0.910	0.905	0.920	0.705
	$\bar{x}$	0.922	0.910	0.920	0.710
	sd	0.025	0.013	0.005	0.013
Isopar M/DCI-4A	1	0.510	0.510	0.505	0.375
	2	0.500	0.510	0.500	0.415
	3	0.510	0.510	0.445*	0.385
	$\bar{x}$	0.507	0.510	0.483	0.392
	sd	0.006	0.000	0.033	0.021

Note: (1) All BOCLE runs performed using Falex Ring.
(2) Winstead balls consistently lower WSD values.
(3) Winstead balls could not differentiate between JP-4 and JP-7.

*Run suspect

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TABLE 16.
SKF TEST BALL COMPOSITION

	*SKF (Swedish)**	*SKF (U.S.)***
Carbon	0.92 — 1.02	0.95 - 1.10
Manganese	0.95 - 1.25	0.25 - 0.45
Silicon	0.50 -- 0.70	0.20 – 0.30
Chromium	0.90 - 1.15	1.30 - 1.60

*% Composition obtained from SKF R&D (U.S.)
**% Composition obtained from Handbook MIL-H1-2 and verified by SKF R&D (U.S.)

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SECTION IV

CONCLUSIONS AND RECOMMENDATIONS

The Falex Ring was shown to significantly enhance BOCLE test precision. Concurrent studies conducted by Rolls Royce, Ltd. in the United Kingdom also produced superior test results. Statistical analysis, performed on data generated by a five member interlaboratory round robin, confirmed a marked improvement in test method repeatability and reproducibility over that realized by the conventional AMS 6444 BOCLE cylinder.

A source for a reliable reproducible AMS 6444 cylinder, yielding the desired precision, does not appear obtainable despite vast effort and expenditure on the part of the lubricity community. It is, therefore, recommended that the CRC be urged to further evaluate the Falex Ring as an alternative to the AMS 6444 material. Advantages for incorporating the Falex Ring into the BOCLE test procedure as the standard test specimen include: definitive, less subjective interpretation of the wear scar; enhanced test precision; and availability at a low cost.

It was shown that the source of the test ball can have a significant effect on BOCLE test results. Most distributors of balls represent a composite of manufacturing sources which is not conducive to the primary goal of standardization of the lubricity test method. SKF precision balls produced in Sweden are manufactured under tight tolerances exhibiting little or no variation from batch to batch. It is recommended that SKF Swedish 12.7 mm (0.5 in.) ball bearings be incorporated into the standard test method. Standardization of all critical test parts and operating parameters is essential in eliminating extraneous variables suspected, or known, to affect test repeatability or reproducibility.

Isopar M + 30 ppm DuPont DCI-4A was shown to be a reproducible fluid suitable for use as a standard reference fluid in the calibration of Falex Rings.

APPENDIX A

ISOPAR M PROPERTY DATA SHEET

TYPICAL PROPERTIES

The values shown here are representative of current production. Some are controlled by manufacturing specifications, while others are not. All of them may vary within modest ranges.

Solvency		**Test Method**
Aniline point, C(F)	89 (192)	ASTM D611
Solubility parameter	7.3	Calculated
Kauri-butanol value	27	ASTM D1133
Volatility		
Flash point, PM, C (F)	80 (176)	ASTM D93
Fire point, COC, C (F)	93 (200)	ASTM D92
Auto-ignition temperature, C (F)	338 (640)	ASTM D286
Flammability limits in air, vol% at 21C (70F)	0.6-6.5	Calculated
Distillation, C (F)		ASTM D86
IBP	207 (405)	
5%	212 (413)	
10%	213 (415)	
50%	223 (434)	
90%	241 (466)	
95%	247 (476)	
Dry point	254 (490)	
FBP	260 (500)	
Vapor pressure, kPa at 38C	4.1	ASTM D2551
Vapor pressure, psia at 100F	0.6	
Composition		
Hydrocarbon type, mass %		
Total saturates	99.5	Mass spectrometer
Aromatics	0.4	UV Analysis
Trace compounds		
Sulfur		
Doctor test	pass	ASTM D484
Total sulfur, ppm	1	Microcoulometer
Peroxides, ppm	<1	Exxon Method
General Properties		
Average molecular weight	191	Cryogenic
Bromine index (1)	230	ASTM D2710
Copper corr., 1/2 hr at BP	2	ASTM D130
Unsulfonated residue, vol%	99+	ASTM D483
UV absorbance		FDA Method
260-319 m	<1.5	21 CFR 172.882

General Properties (cont.)		**Test Method**
320-329m	<0.08	
330-350m	<0.05	
Color, Saybolt	+30	ASTM D156
Color stability, 16 hr at 100C (212F)	+30	
Gravity, API	49.2	ASTM D287
Specific gravity @ 15.6/15.6C	0.784	Calculated
kg/m3	784	
lb/gal	6.53	Calculated
Refractive Index, 20C	1.4362	ASTM D1218
Viscosity		ASTM D445
cp at 25C	2.46	
cp at 100C	0.72	
cSt at 0C	6.80	
cSt at 25C	3.35	
Odor, bulk	very slight	Exxon Method
Odor, residual	none	Exxon Method
Odor stability	excellent	Exxon Method
Freezing point, C (F) <-60 (<-76)		
Specific heat, liquid, kJ/kg/C (Btu/lb/F)		
at 16C (60F)	205 (0.49)	Calculated from
at 66C (150F)	2.26 (0.54)	enthalpy data
at 93C (200F)	2.39 (0.57)	
Heat of vaporization, kJ/kg (Btu/lb)		Est. from Maxwell's "Data Book of
at 100C (212F)	307 (132)	Hydrocarbons" and
at BP	24 (105)	report of API Project 44 (1953)
Surface Properties		
Demulsibility	excellent	Exxon Method
Interfacial tension, dynes/cm at 25C	51.0	ASTM D971
Surface tension dynes/cm at 25C	24.8	du Nuoy
Toxicological Data		
Inhalation, TLV(2) ppm	300(3)	
Acute Oral LD50 (Rat), g/kg	>10	
Acute Dermal LD50 (Rabbit), g/kg	>3.1	

(1) Bromine index = Bromine number × 1000

(2) TLV is a registered trademark of the American Conference of Governmental Industrial Hygienists. It is the threshold limit value or occupational exposure limit the time weighted average concentration for a normal 8-hour workday, 40-hour workweek, to which nearly all workers may be exposed repeatedly without adverse effect. Refer to the most recent Material Safety Data Sheet for the latest recommended maximum exposure limit.

(3) A TLV has not been established for this product. The value shown has been recommended by Exxon Corporation Medical Research based on consideration of available toxicological data. Additional data are being obtained to help define a recommended occupational exposure limit more conclusively.

APPENDIX B

BOCLE MODIFICATION FOR USE WITH FALEX RING

Refer to Figure B-1 when performing the following steps:

1. Remove the six Allen screws that secure the base plate to the top plate.

2. Remove the four Allen screws that secure the load beam pedestal to the top plate.

3. Insert the 1 1/2 X 3 in. shim between the load beam and the top plate. Replace the four Allen securing screws.

4. Reattach the top plate to the base plate.

5. Attach the 3/4 X 3/4 in. shim to the underside of the load beam in such a position that the hydraulic lift plunger meets the shim when in the 'up' position.

   NOTE:

   Contact cement has been found to be satisfactory for attachment of the shim to the load beam.

6. Check the load beam balance and adjust if required.

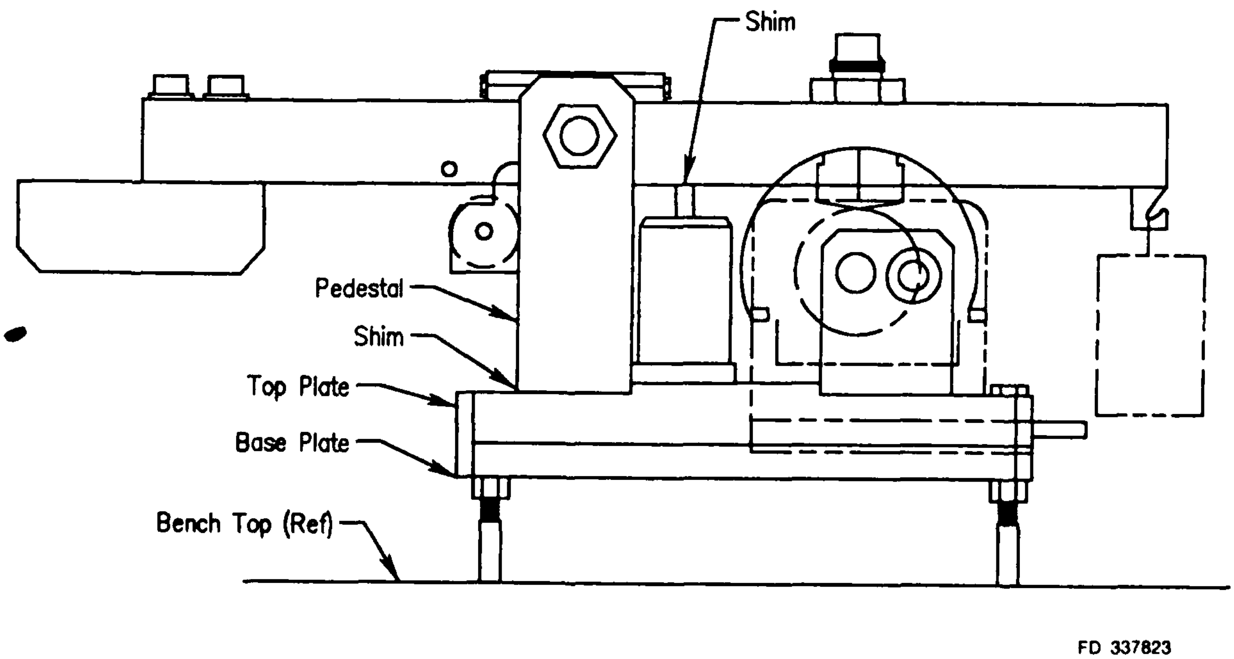

Figure B-1. — BOCLE Modification

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