Institute for
Interlaboratory Studies

Results of Proficiency Test
Level of Contamination
in Lubricants
June 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

During the annual proficiency testing program 2022/2023 the Institute for Interlaboratory Studies (iis) decided to start a separate round robin for the determination of Level of Contamination on Lubricating Oil.

In this first interlaboratory study 9 laboratories in 8 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Level of Contamination proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one sample of used lubricating oil in a 0.5 liter bottle labelled #23085. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

For the preparation of the subsamples a batch of approximately 40 liters of used Hydraulic Oil was used. A defined volume of fresh prepared and well shaken dust suspension of Arizona Dust material in an oil suspension was added to an empty amber glass bottle of 0.5 L by means of a calibrated pipette. The addition was checked by weighing the bottle before and after the addition. In total 30 bottles were prepared and subsequently filled up to 0.5 L from this batch of used Hydraulic Oil and homogenized. The subsamples were labelled #23085.

To each of the participating laboratories one 0.5 L bottle with Hydraulic Oil labelled #23085 was sent on May 10, 2023. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Hydraulic Oil packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine Level of Contamination (counts/mL and ISO4406 scale).

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

$$z_{(target)} = (\text{test result} - \text{average of PT}) / \text{target standard deviation}$$

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

	|z|	< 1	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. One participant reported test results after the final reporting date and one other participant did not report any test results. Not all participants were able to report all tests requested. In total 8 participants reported 45 numerical test results. No outlying test results were observed. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

All data sets were too small to prove if there was a normal Gaussian distribution or not.

4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D7647) and an added designation for the year that the test method was adopted or revised (e.g. D7647:10). When a method has been reapproved an “R” will be added and the year of approval (e.g. D7647:10R18).

Level of Contamination:

Counts/mL: This determination was problematic. No statistical outliers were observed over 3 parameters. All the calculated reproducibilities are not in agreement with the requirements of ASTM D7647:10R18.

ISO4406 scale numbers: This determination was problematic. No statistical outliers were observed over 3 parameters. The calculated reproducibilities for the scale numbers ≥4µm and ≥14µm are not in agreement with the requirements of ASTM D7647:10R18. The test results for ≥6 µm (ISO scale) were not evaluated as the calculated reproducibility was much larger in comparison with the target reproducibility.

Conversion from scale/mL to ISO4406 scale numbers were done correctly.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference methods are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
≥ 4 µm (c)	counts/mL	7	6384	11076	7214
≥ 6 µm (c)	counts/mL	7	2308	4488	1754
≥ 14 µm (c)	counts/mL	7	154	336	208
≥ 4 µm (c)	ISO scale	8	19.5	3.3	1.7
≥ 6 µm (c)	ISO scale	8	17.4	5.2	(1.2)
≥ 14 µm (c)	ISO scale	8	13.1	6.1	2

Table 1: reproducibilities of tests on sample #23085

For results between brackets: no z-scores are calculated.

Without further statistical calculations it can be concluded that for all tests there is not a good compliance of the group of participants with the reference test methods.

4.3 OVERVIEW OF THE PROFICIENCY TEST OF JUNE 2023

	June 2023
Number of reporting laboratories	8
Number of test results	45
Number of statistical outliers	0
Percentage of statistical outliers	0%

Table 2: overview this proficiency test

In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The performance of the determination of the proficiency test was compared to the requirements of the reference test method. The conclusion is given in the following table.

Parameter	June 2023
Counts/mL	--
ISO4406 scal number	--

Table 3: comparison determination to the reference test method

The following performance categories were used:

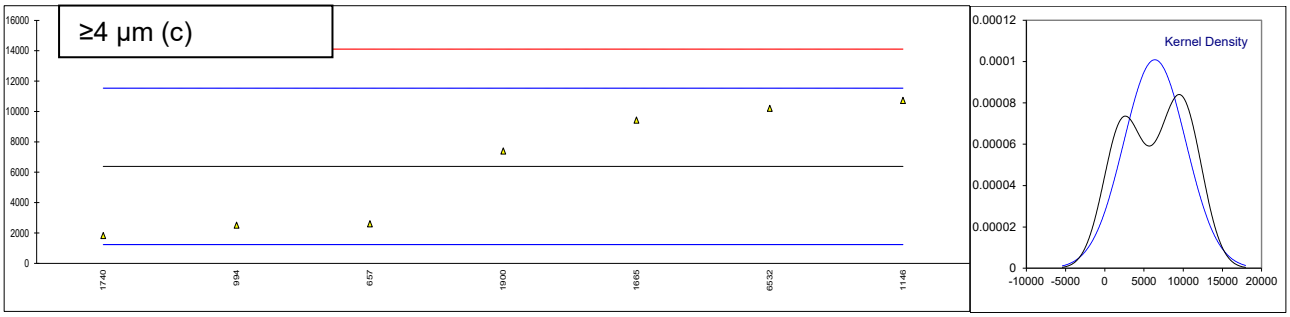
++ : group performed much better than the reference test method.
+ : group performed better than the reference test method.
+/- : group performance equals the reference test method.
- : group performed worse than the reference test method.
-- : group performed much worse than the reference test method.
n.e. : not evaluated.

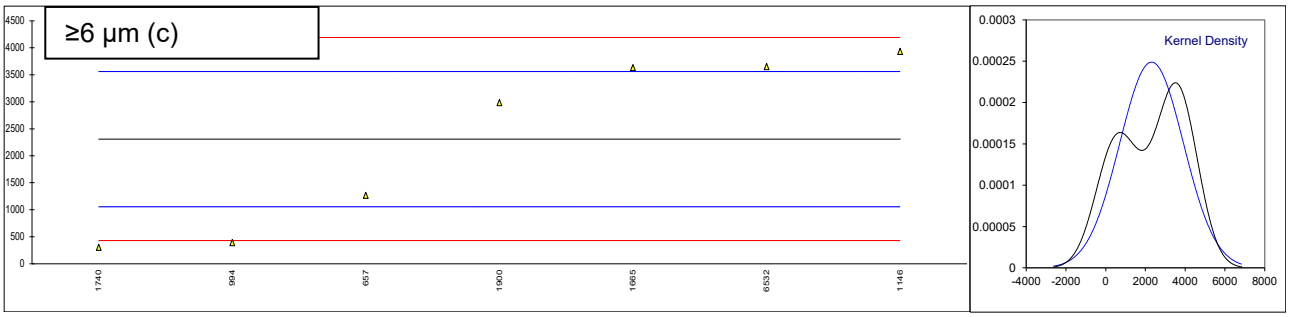
APPENDIX 1

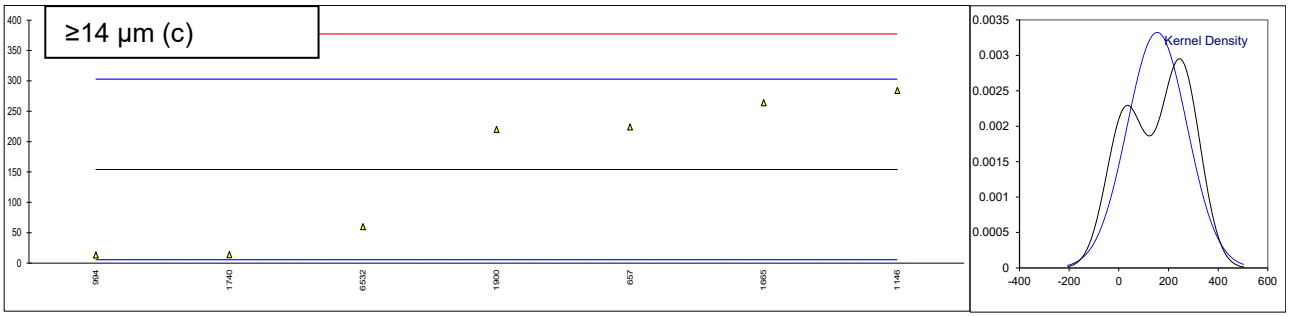
Determination of Level of Contamination on sample #23085; results in counts/mL

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
657	ISO4407	2596	C	-1.47	1266	C	-1.66	224	C	0.94
994	D7647	2514.6		-1.50	390.8		-3.06	13.4		-1.89
1023		-----		-----	-----		-----	-----		-----
1059		-----		-----	-----		-----	-----		-----
1146	In house	10726		1.69	3932		2.59	284		1.75
1665	D7647	9432		1.18	3630		2.11	264		1.48
1740	D7647	1822		-1.77	305		-3.20	14		-1.89
1900	ISO4407	7393		0.39	2984		1.08	220		0.89
6532	ISO4407	10205	C	1.48	3650	C	2.14	60	C	-1.27
	normality	unknown			unknown			unknown		
	n	7			7			7		
	outliers	0			0			0		
	mean (n)	6384.09			2308.26			154.20		
	st.dev. (n)	3955.891			1602.936			120.042		
	R(calc.)	11076.49			4488.22			336.12		
	st.dev.(D7647:10R18)	2576.435			626.527			74.346		
	R(D7647:10R18)	7214.02			1754.28			208.17		

Lab 657: first reported 26 ,13, 2 respectively.
Lab 6532: first reported 102052, 36499, 604 respectively.



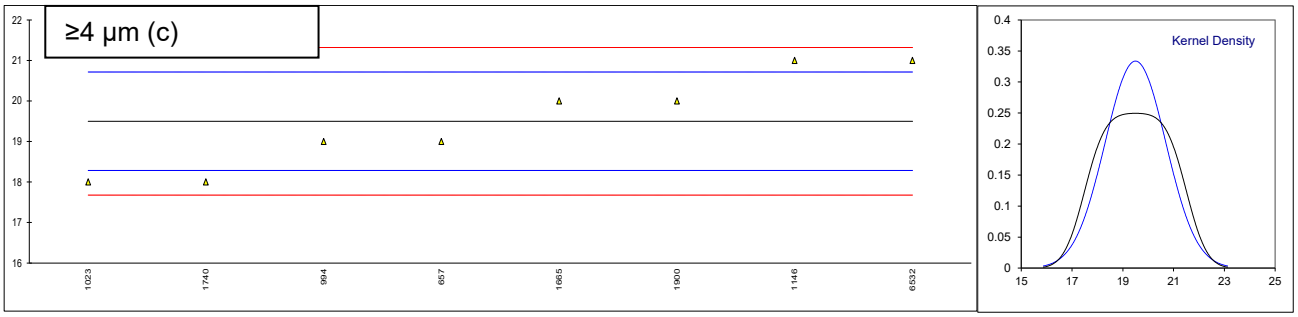


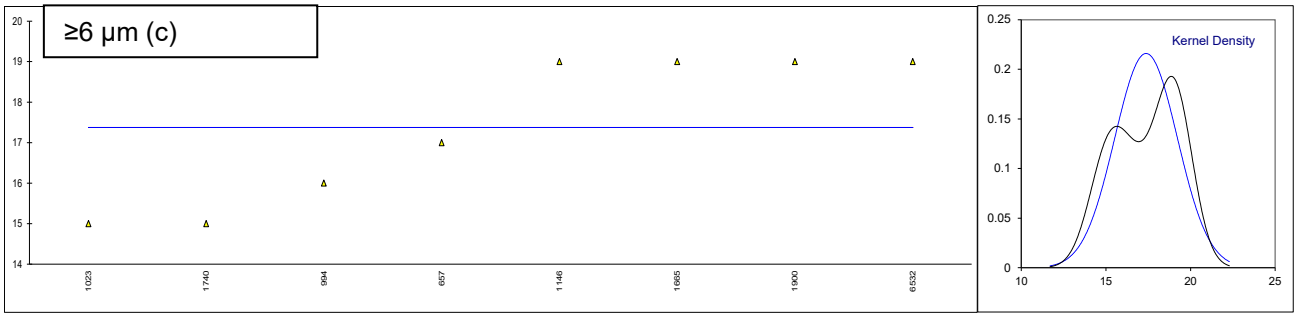


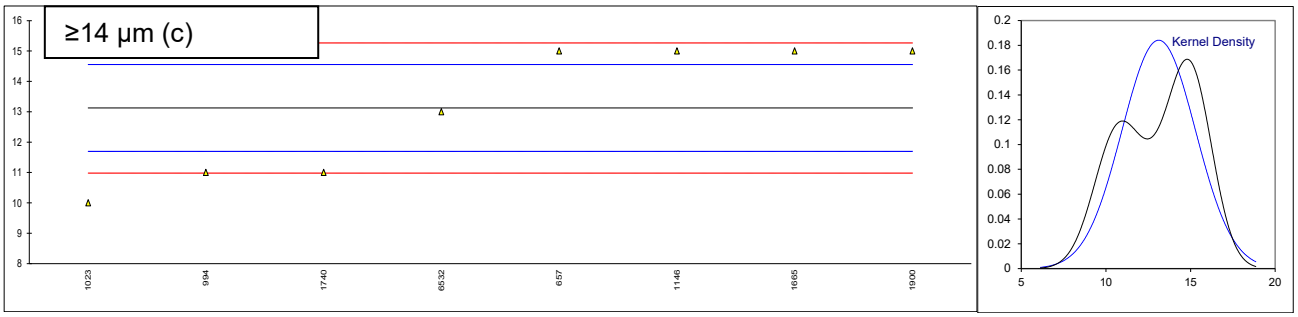
Determination of Level of Contamination on sample #23085; results in ISO4406 scale numbers

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
657	ISO4406	19	C	-0.82	17	C	-----	15	C	2.63
994	D7647	19		-0.82	16		-----	11		-2.98
1023	ISO4406	18		-2.47	15		-----	10		-4.38
1059		-----		-----	-----		-----	-----		-----
1146	In house	21		2.47	19		-----	15		2.63
1665	ISO4406	20		0.82	19		-----	15		2.63
1740	ISO4406	18		-2.47	15		-----	11		-2.98
1900	ISO4406	20		0.82	19		-----	15		2.63
6532	ISO4406	21		2.47	19		-----	13		-0.18
	normality	unknown			unknown			unknown		
	n	8			8			8		
	outliers	0			0			0		
	mean (n)	19.5			17.4			13.1		
	st.dev. (n)	1.19			1.85			2.17		
	R(calc.)	3.3			5.2			6.1		
	st.dev.(D7647:10R18)	0.61			(0.43)			0.71		
	R(D7647:10R18)	1.7			(1.2)			2		

Lab 657: first reported; 18, 16, 12 respectively.







APPENDIX 2

Number of participants per country

1 lab in AZERBAIJAN
1 lab in FRANCE
1 lab in GREECE
1 lab in NETHERLANDS
2 labs in NORWAY
1 lab in SINGAPORE
1 lab in SLOVENIA
1 lab in SPAIN

APPENDIX 3

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

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