

Institute for **Interlaboratory Studies** 

# **Results of Proficiency Test** Turbine Oil (used) May 2022

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

Author:

ing. A. Ouwerkerk

**Correctors:** 

ing. R.J. Starink & ing. M. Meijer ing. A.S. Noordman-de Neef

Approved by:

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#### 1 Introduction

Since 2013 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of used Turbine Oil every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of used Turbine Oil.

In this interlaboratory study 45 laboratories in 32 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the used Turbine Oil 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 Turbine Oil in a 1-liter bottle labelled #22077. 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

A batch of approximately 65 liters of used Turbine Oil was obtained from a local supplier. After homogenization 60 amber glass bottles of 1 L were filled and labelled #22077. The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/L
sample #22077-1	0.88739
sample #22077-2	0.88739
sample #22077-3	0.88738
sample #22077-4	0.88739
sample #22077-5	0.88739
sample #22077-6	0.88738
sample #22077-7	0.88739
sample #22077-8	0.88739

Table 1: homogeneity test results of subsamples #22077

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15 °C in kg/L
r (observed)	0.00001
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #22077

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 1 L bottle of used Turbine Oil labelled #22077 was sent on April 13, 2022. An SDS was added to the sample package.

### 2.5 STABILITY OF THE SAMPLES

The stability of used Turbine 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: Total Acid Number, Color ASTM, Density at 15 °C, Flash Point (C.O.C. and PMcc), Insoluble Color Bodies, Kinematic Viscosity at 40 °C and 100 °C, Viscosity Index, Oxidation Stability RPVOT, Water, Water Separability at 54 °C distilled water and Level of Contamination (counts/mL and ISO4406 scale). Some extra information was asked about the determination of Total Acid Number.

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 F(0.01) for the Rosner's test. Stragglers are marked by F(0.01) for the Dixon's test, by F(0.01) 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_{\text{(target)}} = (test result - average of PT) / target standard deviation
```

The  $z_{\text{(target)}}$  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**

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. One participant reported test results after the extended reporting date and three other participants were not able to report any test results. Not all participants were able to report all tests requested.

In total 42 participants reported 517 numerical test results. Observed were 21 outlying test results, which is 4.1%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

### 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. D2270) and an added designation for the year that the test method was adopted or revised (e.g. D2270:10). When a method has been reapproved an "R" will be added and the year of approval (e.g. D2270:10R16).

<u>Total Acid Number</u>: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D664-A:18e2 BEP and IP at 60 mL, but not with BEP and IP at 125 mL.

When the test results for IP and BEP were evaluated separately, the calculated reproducibilities of the test results are also not in agreement with the end points at 125 mL titration volume.

Remarkably, three participants still used pH 11 for BEP instead of pH 10. In test method ASTM D664:18e2 pH 10 is mentioned.

- <u>Color ASTM</u>: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with ASTM D1500:12R17.
- <u>Density at 15 °C</u>: This determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.
- <u>Flash Point C.O.C.</u>: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D92:18.
- <u>Flash Point PMcc</u>: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D93:20 procedure A. Only three participants used procedure B.
- <u>Insoluble Color Bodies</u>: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7843:21.
- <u>Kinematic Viscosity at 40 °C</u>: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D445:21e1.

Kinematic Viscosity at 100 °C: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D445:21e1.

Viscosity Index: This determination was problematic. No statistical outliers were observed but four test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2270:10R16.

Oxidation Stability RPVOT: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D2272:22.

Water:

This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304:20 procedure B, A and C.

Water Separability at 54 °C, distilled water: This determination was problematic. No statistical outliers were observed over 3 parameters. All calculated reproducibilities are not in agreement with the requirements of ASTM D1401:21.

Level of Contamination: This determination was problematic. Three statistical outliers were observed and five other test results were excluded over 6 parameters. The calculated reproducibilities for the determinations in counts/mL and ISO4406 scale numbers after rejection of the suspect data are not in agreement with the requirements of ASTM D7647:10R18.

#### 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)
Total Acid Number	mg KOH/g	37	0.27	0.10	0.15
Color ASTM		31	4.3	0.8	1
Density at 15 °C	kg/L	33	0.8874	0.0004	0.0005
Flash Point C.O.C.	°C	28	224.1	27.7	18
Flash Point PMcc	°C	29	177.9	10.1	12.6
Insoluble Color Bodies		12	8.4	6.0	8.6
Kinematic Viscosity at 40 °C	mm²/s	37	30.65	0.25	0.22
Kinematic Viscosity at 100 °C	mm²/s	36	5.516	0.085	0.041
Viscosity Index		30	118.0	3.8	2

Parameter	unit	n	average	2.8 * sd	R(lit)
Oxidation Stability RPVOT	minutes	13	929	263	213
Water	mg/kg	35	174.0	59.3	193.7
Water Separability at 54 °C, distilled	ed water				
Time ≤ 3 mL emulsion	minutes	8	24.4	30.2	20
Time 37 mL water	minutes	7	27.8	37.1	20
Time to complete break	minutes	5	22.8	31.9	20
Level of Contamination					
≥ 4 µm (c)	counts/mL	18	1314	1787	1485
≥ 6 µm (c)	counts/mL	20	168	264	127
≥ 14 µm (c)	counts/mL	20	14	23	19
≥ 4 µm (c)	ISO scale	20	17	3	2
≥ 6 µm (c)	ISO scale	22	14	3	1
≥ 14 µm (c)	ISO scale	22	11	4	2

Table 3: reproducibilities of tests on sample #22077

Without further statistical calculations, it can be concluded that for many tests there is not a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2022 WITH PREVIOUS PTS

	May 2022	May 2021	May 2020	May 2019	May 2018
Number of reporting laboratories	42	45	35	42	43
Number of test results	517	547	440	600	581
Number of statistical outliers	21	24	25	13	10
Percentage of statistical outliers	4.1%	4.4%	5.7%	2.2%	1.7%

Table 4: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency tests was compared to the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	May 2022	May 2021	May 2020	May 2019	May 2018
Total Acid Number	+	+/-	-	+	
Color ASTM	+	-	+	+	+
Density at 15 °C	+	+/-	+/-	+	+
Flash Point C.O.C.	-	-	+/-	+/-	-
Flash Point PMcc	+	+	+	+	+
Insoluble Color Bodies	+	+	+	+/-	+
Kinematic Viscosity at 40 °C	-	+	-	-	+
Kinematic Viscosity at 100 °C		-		-	+/-

Parameter	May 2022	May 2021	May 2020	May 2019	May 2018
Viscosity Index	-	-		-	()
Oxidation Stability RPVOT	-			()	-
Water	++	-	++	++	++
Water Separability	-	++	+	+	++
Level of Contamination	-			()	-

Table 5: comparison determinations to the reference test methods

For results between brackets: no z-scores are calculated

### 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

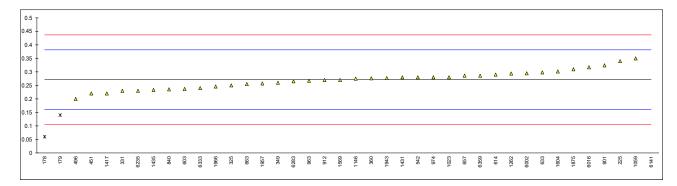
-- : group performed much worse than the reference test method

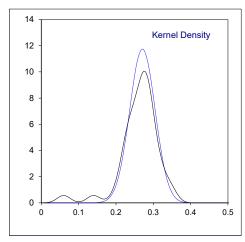
n.e. : not evaluated

### **APPENDIX 1**

Determination of Total Acid Number on sample #22077; results in mg KOH/g

Deterr	nination of Total Acid Num	per on s	sampie #	-22077	; results in mg KOH/	g	
lah	method	value	mark	z(targ)	end point determination	vol. titration solvent re	emarks
	D664-A	0.06	R(0.01)	-3.81			,,,,,,,,,
	D664-A	0.14	R(0.05)	-2.37		60 mL	
	D974	0.34		1.25			
237	D664-A	<0.1		<-3.09	Inflection Point	125 mL fa	ılse -?
325	D664-A	0.25		-0.38	Buffer End Point pH 10	125 mL	
331	D664Mod.	0.23			Inflection Point	60 mL	
	D664-A	0.26			Inflection Point	125 mL	
	D664-A	0.276			Inflection Point	60 mL	
	D004-A			0.03			
432	D004 A						
451		0.22			Buffer End Point pH 10	60 mL	
	D664-A	0.20			Buffer End Point pH 10	60 mL	
542	D974	0.28		0.16			
603	D664-A	0.237		-0.61	Inflection Point	125 mL	
614	D664-A	0.29		0.34		60 mL	
633	D664-A	0.29815		0.49	Inflection Point	125 mL	
	D664-A	0.286			Inflection Point	60 mL	
	D664-A	0.255			Buffer End Point pH 10	60 mL	
704	D004-A			-0.23	Ballet Ella i ollit pri 10		
	D664-B	0.235			Buffer End Point pH 10	 60 ml	
	D004-D					60 mL	
862	5004.4						
901	D664-A	0.324		0.96			
	D974	0.27		-0.02			
962							
963	D664-B	0.267		-0.07	Inflection Point	60 mL	
974	D664-A	0.28		0.16	Inflection Point	125 mL	
	D8045	0.28		0.16			
	ISO6619	0.35			Buffer End Point pH 11	60 mL	
	D664-A	0.275			Buffer End Point pH 10	125 mL	
	ISO6618	0.273		0.07		60 mL	
	D664-A	0.220			Buffer End Point pH 10	60 mL	
	D664-A	0.2798			Inflection Point	60 mL	
	D664-A	0.233			Buffer End Point pH 10	100 ml	
1569	D664-A	0.27			Inflection Point	125 mL	
1604	D664-A	0.3016		0.55	Inflection Point	60 mL	
1875	ISO6618	0.3097		0.70			
1943	ISO6618	0.277		0.11			
	D664-A	0.257		-0.25	Buffer End Point pH 11	125 mL	
	ISO6618	0.2457		-0.46			
	D664-A	0.295			Inflection Point	60 mL	
	D664-A	0.233			Inflection Point	60 mL	
			D(0.01)				
	D974	1.11	R(0.01)	15.16			
	D664-A	0.230			Inflection Point	60 mL	
	D664-A	0.265			Buffer End Point pH 11	60 mL	
	D8045	0.24		-0.56	Inflection Point	60 mL	
6359	D664-A	0.286		0.27		60 mL	
					Buffer End Point only	Inflection Point only	
	normality	OK			suspect	OK	
	n	37			15	15	
	outliers	3			0	1	
	mean (n)	0.2709			0.2580	0.2712	
	st.dev. (n)	0.03399			0.03707	0.02724	
	R(calc.)	0.03399			0.1038	0.02724	
	st.dev.(D664-A:18e2 BEP, 60mL)						
	, ,	0.05534			0.05276		
Ca	R(D664-A:18e2 BEP, 60mL)	0.1550			0.1477		
Compa		0.0004			0.0700		
	R(D664-A:18e2 BEP, 125mL)	0.0801			0.0760		
	R(D664-A:18e2 IP, 60mL)	0.1379				0.1380	
	R(D664-A:18e2 IP, 125mL)	0.0560				0.0561	

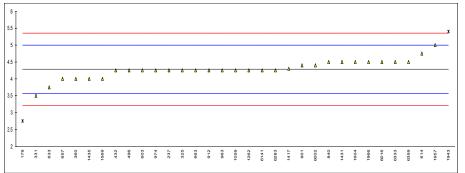


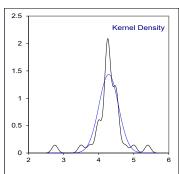


# Determination of Color ASTM on sample #22077;

lab	method	reported test result	iis conversion *)	mark	z(targ)	remarks
178						
	D1500	L3.0	2.75	R(0.01)	-4.30	
225						
	D1500	L4.5	4.25		-0.10	
	D6045	L4.5	4.25		-0.10	
	D1500	3.5	3.5	С	-2.20	first reported 3
349						
	D1500	4.0	4.0		-0.80	
	D1500	L4,5	4.25		-0.10	
451						
	D1500	L4.5	4.25		-0.10	
542						
	D1500	L4.5	4.25		-0.10	
	D1500	<5.0	4.75		1.30	
	D1500	L4.0	3.75	С	-1.50	first reported 3.5
	D1500	4.0	4.0		-0.80	
	D1500	L4.5	4.25		-0.10	
704						
	D1500	4.5	4.5		0.60	
862						
	D1500	4.4	4.4		0.32	
912	D1500	<4.5	4.25		-0.10	
962						
963	D1500	L4.5	4.25		-0.10	
	D1500	L4.5	4.25		-0.10	
1023						
	D1500	L4,5	4.25		-0.10	
1146						
	ISO2049	L 4.5	4.25		-0.10	
1417	D6045	4.3	4.3		0.04	
	D1500	4.5	4.5		0.60	
	D1500	4.0	4.0		-0.80	
	D1500	4.0	4.0		-0.80	
	D1500	4.5	4.5		0.60	
1875						
1943	ISO2049	5.4	5.4	R(0.05)	3.12	
1957	D1500	5.0	5.0		2.00	
	D1500	4.5	4.5		0.60	
6002	In house	4.4	4.4		0.32	
	D1500	4.5	4.5		0.60	
6141	D1500	L4.5	4.25		-0.10	
6235						
6283	D1500	L4.5	4.25		-0.10	
6333	D1500	4.5	4.5		0.60	
6359	D1500	4.5	4.5		0.60	
	194		. 01/			
	normality		not OK			
	n		31			
	outliers		2			
	mean (n)		4.29			
	st.dev. (n)		0.278			
	R(calc.)		0.78			
	st.dev.(D1500:12R17)		0.357			
	R(D1500:12R17)		1			

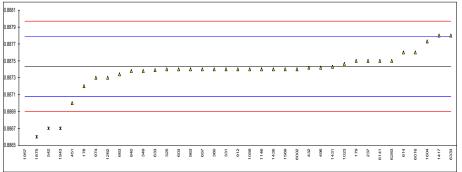
<sup>\*)</sup> In the calculation of the mean, standard deviation and the reproducibility in this column, a reported value of 'L y' is changed tot y-0.25 (for example L5.5 into 5.25).

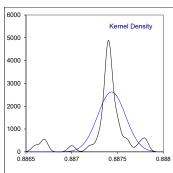




# Determination of Density at 15 °C on sample #22077; results in kg/L

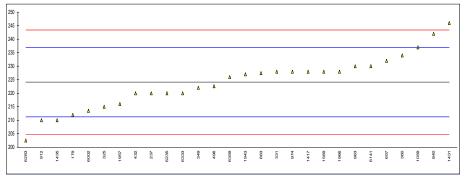
lab	method	value	mark	z(targ)	remarks
178	D4052	0.8872		-1.32	
179	D4052	0.8875		0.36	
225					
237	D4052	0.8875	С	0.36	first reported 888.6 kg/m <sup>3</sup>
325	D4052	0.8874		-0.20	
331	ISO12185	0.8874		-0.20	
349	D4052	0.88738		-0.31	
360	D4052	0.8874		-0.20	
432	ISO12185	0.88742		-0.08	
	D4052	0.8870		-2.44	
496	ISO12185	0.88742		-0.08	
	D7042	0.8867	C,R(0.01)		first reported 887 kg/m <sup>3</sup>
	D4052	0.8874		-0.20	
	D4052	0.8876		0.92	
	D4052	0.88739		-0.25	
	D4052	0.8874		-0.20	
	D4052	0.88734		-0.53	
704	D.4050	0.00700			
	D4052	0.88738		-0.31	
862					
901	D40E2	0.0074		0.20	
962	D4052	0.8874		-0.20	
	D4052	0.8874		-0.20	
	D4052 D4052	0.8873		-0.20	
	D4052 D4052	0.887465		0.17	
	ISO12185	0.8874		-0.20	
	D4052	0.8874		-0.20	
	D4052	0.8873		-0.76	
	IP365	0.8878	С	2.04	
	ISO12185	0.88743		-0.03	
	D4052	0.8874		-0.20	
1569	ISO12185	0.8874		-0.20	
	D1298	0.88773		1.65	
1875	D51757	0.8866	R(0.01)	-4.68	
	ISO3675	0.8867	C,R(0.01)	-4.12	first reported 886.9 kg/m <sup>3</sup>
	D4052	0.8862	R(0.01)	-6.92	
1966			W		test result withdrawn, reported 886.83 kg/m³
	ISO12185	0.8874		-0.20	
	D4052	0.8876		0.92	
	D4052	0.8875		0.36	
6235	D.4050	0.0075			
	D4052	0.8875	0	0.36	5
	D7042	0.8878	C		first reported 0.8878
6359			W		test result withdrawn, reported 0.8878
	normality	not OK			
	n	33			
	outliers	4			
	mean (n)	0.88744			
	st.dev. (n)	0.000152			
	R(calc.)	0.00043			
	st.dev.(ISO12185:96)	0.000179			
	R(ISO12185:96)	0.0005			
	•				

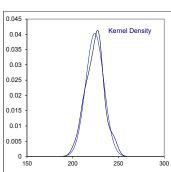




# Determination of Flash Point C.O.C. on sample #22077; results in °C

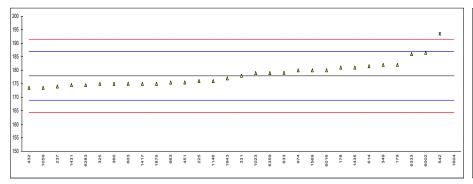
lab	method	value	mark	z(targ)	remarks
178					
179	D92	212		-1.88	
225					
	D92	220		-0.64	
	D92	215		-1.42	
	D92	228		0.61	
	D92	222		-0.33	
	ISO2592 D92	234 220		1.54 -0.64	
451	D9Z			-0.04	
496	D92	222.6		-0.23	
542	D02				
603					
614					
633					
657	D92	232		1.23	
663	D92	227.4	С	0.51	first reported 175.48
704					
	D92	242		2.78	
862					
901					
	D92	210		-2.19	
962	D00				
	D92	230.0		0.92	
1023	D92	228		0.61	
1023	ISO2592	237		2.01	
1146	1002002				
1262					
1417	D92	228		0.61	
1431		246	С	3.41	first reported 252
1435		210.0		-2.19	•
1569		228		0.61	
1604					
1875					
	ISO2592	227		0.45	
1957		216		-1.26	
1966		228		0.61	
	ISO2592	213.5		-1.65	
6016 6141	DOS	220		0.02	
	ISO2592	230 220		0.92	
6283		220 202.5		-0.64 -3.36	
6333		202.5	С	-0.64	first reported 266
6359		226	0	0.29	mot reported 200
0000	202			0.20	
	normality	OK			
	n	28			
	outliers	0			
	mean (n)	224.11			
	st.dev. (n)	9.890			
	R(calc.)	27.69			
	st.dev.(D92:18)	6.429			
	R(D92:18)	18			

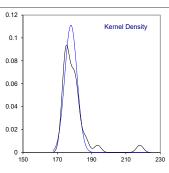




# Determination of Flash Point PMcc on sample #22077; results in °C

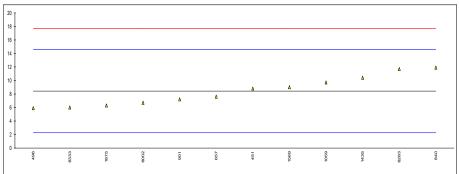
lab	method	value	mark	z(targ)	remarks
178		181		0.68	
	D93-A	182.0		0.90	
	D93-B	176.0		-0.43	
	D93-B	174.0		-0.87	
	D93-A	175.0		-0.65	
	D93-A	173.0		0.01	
	D93-A	182		0.01	
	ISO2719-A	175.0		-0.65	
	D93-A	173.5		-0.98	
	D93-A	175.5		-0.54	
496	D=00.4		0.5/0.04		n
	D7094	193.5	C,R(0.01)	3.45	first reported 195
	D93-A	175.0		-0.65	
	D93-A	181.5		0.79	
633	D93-A	179.075		0.25	
657					
663	D93-A	175.48	С	-0.54	first reported 227.4
704					
840					
862					
901					
912					
962					
963					
	D93-A	180		0.46	
	D93-A	179.0		0.40	
	ISO2719-A	173.5		-0.98	
		175.5			
1262	D93-A			-0.43	
	D02 A	 175		0.65	
1417		175		-0.65	
	D93-A	174.5		-0.76	
	D93-A	181.0		0.68	
	D93-A	180		0.46	
	D93-A	218.1	R(0.01)	8.90	
1875	ISO2719-B	175		-0.65	
1943	ISO2719-A	177		-0.21	
1957					
1966					
6002	ISO2719-A	186.5		1.90	
6016	D93-A	180		0.46	
6141					
6235					
	D93-A	174.5		-0.76	
	D7094	186		1.79	
	D93-A	179		0.24	
0000	DOO'A	175		0.24	
	normality	OK			
	•				
	n	29			
	outliers	2			
	mean (n)	177.93			
	st.dev. (n)	3.591			
	R(calc.)	10.06			
	st.dev.(D93-A:20)	4.512			
	R(D93-A:20)	12.63			

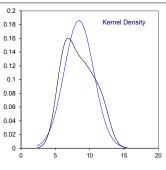




# Determination of Insoluble Color Bodies, membrane patch colorimetry on sample #22077;

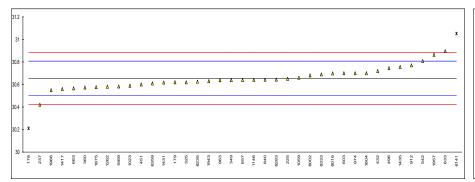
lab	method	value	mark z(targ)	remarks
178				
179				
225				
237				
325				
331				
349				
360				
432				
	D7843	8.8	0.12	
	D7843	5.9	-0.82	
542				
603				
614				
633				
	D7843	7.6	-0.27	
663				
704	D7040	44.0	4.40	
	D7843	11.9	1.12	
862	D7042	 7.0	0.40	
901	D7843	7.2	-0.40	
912 962				
963				
974				
1023				
	D7843	9.7	0.41	
1146	D7040			
1262				
1417				
1431				
	D7843	10.4	0.64	
1569		9	0.18	
1604				
	D7843	6.3	-0.69	
1943				
1957				
1966				
	D7843	6.7	-0.56	
6016				
6141				
6235	D=0.40			
	D7843	11.7	1.06	
6333	D7843	6	-0.79	
6359				
	n armality	OK		
	normality	OK		
	n outliere	12		
	outliers mean (n)	0 8.43		
		8.43 2.147		
	st.dev. (n) R(calc.)	6.01		
	st.dev.(D7843:21)	3.087		
	R(D7843:21)	8.64		
	11(070.21)	0.04		

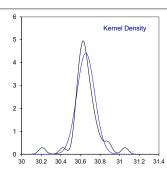




# Determination of Kinematic Viscosity at 40 °C on sample #22077; results in mm<sup>2</sup>/s

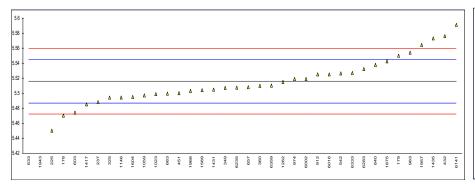
lab	method	value	mark	z(targ)	remarks
178	D7279 corrected to D445	30.21	R(0.01)	-5.77	
	D445	30.62	, ,	-0.44	
225	D445	30.65		-0.05	
237	D445	30.42		-3.04	
	D445	30.62		-0.44	
331					
	D445	30.64		-0.18	
	D445	30.573		-1.05	
	ISO3104	30.72		0.86	
	D7279 corrected to D445	30.6		-0.70	
496	D445	30.745		1.18	
542	D7042	30.8085		2.00	
603	D7042	30.70		0.60	
614					
633	D445	30.896		3.14	
657	D445	30.64		-0.18	
663	D445	30.566		-1.15	
704					
840	D445	30.643		-0.14	
862					
901					
912	D445	30.77		1.50	
962					
963	D445	30.638		-0.21	
	D445	30.70		0.60	
	D445	30.5890		-0.85	
	ISO3104	30.66		0.08	
	D445	30.641		-0.17	
	ISO3104	30.58		-0.96	
	D445	30.56		-1.22	
	D7042	30.6175		-0.48	
	D7042	30.756		1.32	
	D445	30.583		-0.92	
	D445	30.70		0.60	
	D7042	30.576		-1.02	
	ISO3104	30.63		-0.31	
	D7042	30.864		2.73	
	D445	30.549		-1.37	
	ISO3104	30.681		0.35	
	D7042	30.699	D(0.04)	0.58	
	D445	31.0529	R(0.01)	5.18	
	ISO3104	30.625		-0.38	
	D7042	30.644		-0.13	
	D7042	30.69		0.47	
0339	D445	30.61		-0.57	
	normality	suspect			
	n	37			
	outliers	2			
	mean (n)	30.6542			
	st.dev. (n)	0.09040			
	R(calc.)	0.05040			
	st.dev.(D445:21e1)	0.07698			
	R(D445:21e1)	0.2155			
	· · · · · · · · · · · · · · · · · · ·	J			

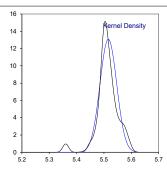




# Determination of Kinematic Viscosity at 100 °C on sample #22077; results in mm<sup>2</sup>/s

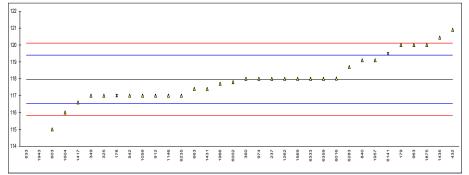
lab	method	value	mark	z(targ)	remarks
	D7279 corrected to D445	5.47		-3.15	
	D445	5.55		2.33	
225	D445	5.450		-4.52	
	D445	5.488		-1.92	
	D445	5.494		-1.51	
331					
349	D445	5.507		-0.62	
360	D445	5.5097		-0.43	
432	ISO3104	5.576		4.11	
451	D7279 corrected to D445	5.50		-1.10	
496					
542	D7042	5.5263		0.71	
603	D7042	5.474	С	-2.88	first reported 5.465
614					
633	D445	5.161	C,R(0.01)	-24.31	first reported 5.1852
	D445	5.508		-0.55	
	D445	5.4995		-1.13	
704					
	D445	5.5376		1.48	
862					
901					
	D445	5.525		0.62	
962					
	D445	5.554		2.60	
	D445	5.519		0.21	
	D445	5.4987		-1.18	
	ISO3104	5.497		-1.30	
	D445 ISO3104	5.4941		-1.50	
	D445	5.515 5.485		-0.07 -2.12	
	D7042	5.465		-2.12 -0.77	
	D7042 D7042	5.573		3.90	
	D445	5.504		-0.82	
	D445	5.495		-1.44	
	D7042	5.5424		1.81	
	ISO3104	5.36	C,R(0.01)	-10.68	first reported 5.38
	D7042	5.5643	0,11(0.01)	3.31	mot reported 0.00
	D445	5.503		-0.89	
	ISO3104	5.519		0.21	
	D7042	5.525		0.62	
	D445	5.5911		5.14	
6235	ISO3104	5.5075		-0.58	
	D7042	5.5322		1.11	
6333	D7042	5.527		0.75	
6359	D445	5.510		-0.41	
		OK			
	normality	OK			
	n outliere	36			
	outliers	2 5 5160			
	mean (n)	5.5160			
	st.dev. (n)	0.03050			
	R(calc.) st.dev.(D445:21e1)	0.0854 0.01461			
	R(D445:21e1)	0.01401			
	(2110.2101)	3.0 100			

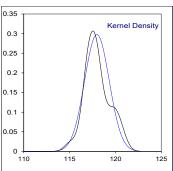




# Determination of Viscosity Index on sample #22077;

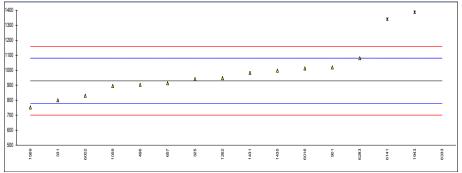
lab	method	value	mark	z(targ)	remarks
178	D2270	117	ex,E	-1.36	test result excluded as statistical outlier in KV 40 °C, iis calc. 118
	D2270	120	,	2.84	,
225					
237	D2270	118		0.04	
	D2270	117		-1.36	
331					
349	D2270	117		-1.36	
360	ISO2909	118.0		0.04	
432	ISO2909	120.9		4.10	
451					
496					
	D2270	117		-1.36	
	D2270	115	С	-4.16	first reported 114
614					
	D2270	93.5	C,ex,E	-34.26	fr. 95.341, test result excluded as statist. outlier in KV 100 °C, iis calc. 95
657					
	D2270	117.4		-0.80	
704	D0070	440.4		4.50	
	D2270	119.1		1.58	
862					
901	D2270	117		1.26	
912	D2270	117 		-1.36	
	D2270	120		2.84	
	D2270 D2270	118		0.04	
1023	D2210				
	ISO2909	117		-1.36	
	D2270	117		-1.36	
	ISO2909	118		0.04	
	D2270	116.6		-1.92	
	D2270	117.4		-0.80	
	D2270	120.441		3.46	
1569	D2270	118		0.04	
1604	D2270	116		-2.76	
1875	ISO2909	120		2.84	
	ISO2909	108.2	C,ex	-13.68	fr. 109.7, test result excluded as statistical outlier in KV 100 °C
1957	D2270	119.1		1.58	
	D2270	117.71		-0.37	
	ISO2909	117.8		-0.24	
	D2270	118.013		0.06	
	D2270	119.484	ex		test result excluded as statistical outlier in KV 40 °C
	ISO2909	117		-1.36	
	D2270	118.7		1.02	
	D2270 D2270	118		0.04	
0339	D2270	118		0.04	
	normality	OK			
	n	30			
	outliers	0+4ex			
	mean (n)	117.97			
	st.dev. (n)	1.340			
	R(calc.)	3.75			
	st.dev.(D2270:10R16)	0.714			
	R(D2270:10R16)	2			

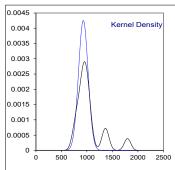




# Determination of Oxidation Stability RPVOT on sample #22077; results in minutes

178	lab	method	value	mark	z(targ)	remarks
179	178					
237	179					
325 D2272-B 941 0.16 331 D2272-B 800 -1.69 349	225					
331 D2272-B 800 -1.69 349 349 349 349 340 341 342 345 347 348 348 349 349 349 349 349 349 349 349 349 349	237					
349	325	D2272-A	941		0.16	
380	331	D2272-B	800		-1.69	
432 451 456 D2272-B 903 -0.34 542	349					
451 496 D2272-B 903						
496 D2272-B 903 -0.34 542						
542						
603 614 614 633 667 D2272-B 913		D2272-B	903		-0.34	
634						
633						
663						
663 704 840 862						
704 840 840 840 840 852 901 D2272-A 1019 1.19 912 962 963 974 1023 1059 D2272-B 895 1414 1407 1431 D2272-B 983 0.72 1435 D2272-B 1386 DG(0.01) 6.01 1957 1966 1966 1966 002 D2272-B 830 -1.30 6016 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-A 1340 DG(0.01) 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-A 1340 DG(0.01) 1.11 6359  normality OK n 0Utilers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7		D2272-B	913		-0.21	
840 862 901 D2272-A 1019 1.19 912 963 974 1023 1059 D2272-B 895 -0.44 1146 1262 D2272-A 947 0.24 1417 1431 D2272-B 983 0.72 1435 D2272-A 997 0.90 1569 D2272-B 983 0.72 1435 D2272-B 983 0.72 1435 D2272-B 983 0.72 1435 D2272-B 983 0.72 1437 D2272-B 983 0.72 1438 D2272-B 1386 DG(0.01) 6.01 1957 1966 1968 0002 D2272-B 830 -1.30 6016 D2272-A 1340 DG(0.01) 5.41 D2272-A 1340 DG(0.01) 5.41 D2272-A 1340 DG(0.01) 5.41 D2272-A 1340 DG(0.01) 5.41 D2272-A 1340 DG(0.01) 1.10 6141 D2272-A 1340 DG(0.01) 5.41 G235 G235 G283 D2272-B 1796 G(0.01) 1.10 6141 D2272-A 1340 DG(0.01) 1.11 6141 D2272-A 1340 DG(0.01) 1.141 6359						
862 901 D2272-A 1019 1.19 912 962 974 1023 1059 D2272-B 895 1431 D2272-A 997 1431 D2272-B 983 0.72 1435 D2272-A 997 0.90 1569 D2272-B 1386 D2272-B 1386 D2272-B 1386 D2272-B 1386 D2272-B 1386 D2272-B 1386 D380 1966						
901 D2272-A 1019 1.19 912 962 963 974 1023 1059 D2272-B 895 -0.44 1146 1262 D2272-A 947 0.24 1417 1431 D2272-B 983 0.72 1435 D2272-A 997 0.90 1569 D2272-B 752 -2.32 1604 1875 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 6002 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-A 1080 1.99 6333 D2272-B 1796 G(0.01) 11.41 6236  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7						
912 962 974		D0070 A				
962 963 974 1023 1059 D2272-B 895 -0.44 1146 1262 D2272-A 947 0.24 1417 1431 D2272-B 983 0.72 1435 D2272-A 997 0.90 1569 D2272-B 752 -2.32 1604 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 0002 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1020 D6(0.01) 5.41 6235 6283 D2272-A 1080 01.99 6333 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7		D2272-A	1019		1.19	
963 974 1023 1059 D2272-B 895 -0.44 1146 1262 D2272-A 947 0.24 1431 D2272-B 983 0.72 1435 D2272-B 983 0.72 1435 D2272-B 97 0.90 1569 D2272-B 752 -2.32 1604 1875 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 6002 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1012 1.10 6141 D2272-A 1080 DG(0.01) 5.41 6235 6233 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7						
974 1023 1059 D2272-B 895 -0.44 1146 1262 D2272-A 947 0.24 1417 1431 D2272-B 983 0.72 1435 D2272-A 997 0.90 1569 D2272-B 752 -2.32 1604 1875 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 1966 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-A 1080 1.99 6333 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 926.7						
1023 1059 D2272-B 895 -0.44 1146 1262 D2272-A 947 0.24 1417 1431 D2272-B 983 0.72 1435 D2272-A 997 0.90 1569 D2272-B 752 -2.32 1604 1875 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 6002 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-B 1080 1.99 6333 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 926.7	963					
1059 D2272-B						
1146 1262 D2272-A 1417		D0070 D				
1262 D2272-A 947 0.24 1417 1431 D2272-B 983 0.72 1435 D2272-A 997 0.90 1569 D2272-B 752 -2.32 1604 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 1966 1960 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-B 1796 G(0.01) 11.41 6359		D2272-B				
1417 1431 D2272-B 1435 D2272-A 1435 D2272-B 1569 D2272-B 1752 1604 1875 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 6002 D2272-B 830 6016 D2272-A 1012 6141 D2272-A 1012 6141 D2272-A 1012 6141 D2272-A 1080 6033 D2272-B 1080 6033 D2272-B 1796 G(0.01) 11.41 6359  normality NK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 2027-A 1080 078 078 078 078 078 078 078 078 078		D0070 A				
1431 D2272-B 983 0.72 1435 D2272-A 997 0.90 1569 D2272-B 752 -2.32 1604 1875 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 6002 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-B 1796 G(0.01) 11.41 6339 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7		D2212-A				
1435 D2272-A 997 0.90 1569 D2272-B 752 -2.32 1604 1875 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 6002 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-A 1080 1.99 6333 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7		D0070 D				
1569 D2272-B			983			
1604 1875 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 6002 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-A 1080 1.99 6333 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7						
1875 1943 D2272-B 1386 DG(0.01) 6.01 1957 1966	1604	D2212-D				
1943 D2272-B 1386 DG(0.01) 6.01 1957 1966 6002 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-A 1080 1.99 6333 D2272-B 1796 G(0.01) 11.41 6359						
1957		D2272 B		DG(0.01)		
1966		DZZ1Z-D		DG(0.01)		
6002 D2272-B 830 -1.30 6016 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235						
6016 D2272-A 1012 1.10 6141 D2272-A 1340 DG(0.01) 5.41 6235 6283 D2272-A 1080 1.99 6333 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7		D2272-B				
6141 D2272-A 1340 DG(0.01) 5.41 6235						
6235				DG(0.01)		
6283 D2272-A 1080 1.99 6333 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7				20(0.01)		
6333 D2272-B 1796 G(0.01) 11.41 6359  normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7		D2272-A				
normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7				G(0.01)		
normality OK n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7		DEETE D		G(0.01)		
n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7	5000					
n 13 outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7		normality	OK			
outliers 3 mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7						
mean (n) 928.6 st.dev. (n) 93.83 R(calc.) 262.7						
st.dev. (n) 93.83 R(calc.) 262.7						
R(calc.) 262.7		st.dev. (n)				
et dev (D2272:22) 76.04		R(calc.)				
31.UGV.(DZZ1Z.ZZ) / U.U4		st.dev.(D2272:22)	76.04			
R(D2272:22) 212.9		R(D2272:22)	212.9			
		,				





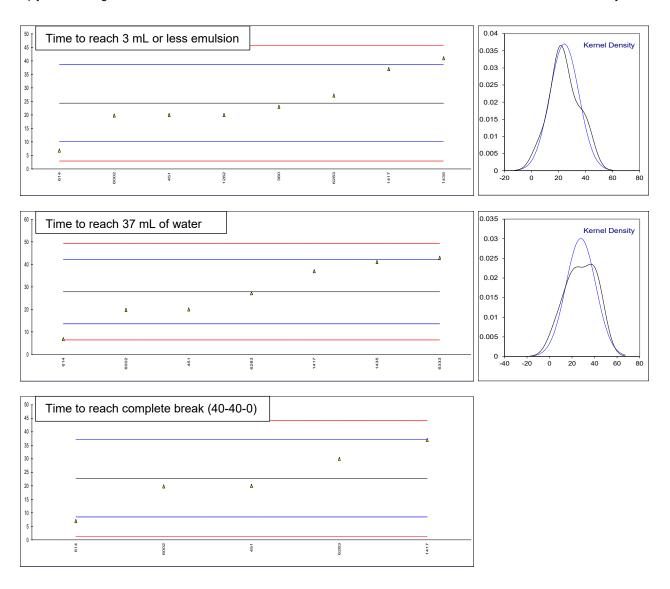
# Determination of Water on sample #22077; results in mg/kg

lak	mathad	volue	mark =/taun)	romorko	
lab	method	value	mark z(targ)	remarks	
178	D6304-C:20	136	-0.55		
179	D6304-C:20	173	-0.01		
225					
237	D6304-C:16e1	210	0.52		
	D6304-C:20	172	-0.03		
331		176	0.03		
	D6304-A:16e1	188	0.20		
	ISO12937	198.0	0.35		
	D6304-B:20	175.58	0.02		
451					
496	D6304-B:20	150	-0.35		
542	D6304-A:20	160.9	-0.19		
603					
614	D6304-B:20	159	-0.22		
633		190.16	0.23		
657	D6304-C:16e1	177.2	0.05		
663	B0004 0.1001				
704					
	D6304 B:30	100			
840	D6304-B:20	123	-0.74		
862					
901	D6304-A:20	185.7	0.17		
912	D6304-B:20	175	0.01		
962					
963	D6304-B	199.2	0.36		
974					
1023	D6304-A:20	189.9	0.23		
1059	_ 000 . / 1.20		0.20		
	D6304-B:20	170	-0.06		
	ISO760				
		155	-0.27		
1417		198	0.35		
1431		217.61	0.63		
	D6304-A:20	192	0.26		
1569	D6304-B:16e1	182	0.12		
1604	D6304-B:16e1	187.5	0.20		
1875	ISO12937	170	-0.06		
	EN60814	125.04	-0.71		
1957		160.8	-0.19		
1966		172.27	-0.02		
	In house		-0.02		
		171.98			
6016	D6304-A:20	185.6	0.17		
6141					
6235		172	-0.03		
6283		178.4	0.06		
6333	D6304-B:20	161	-0.19		
6359	D6304-A:20	152	-0.32		
	normality	OK			
	n	35			
	outliers	0			
	mean (n)	174.00			
	` '	21.161			
	st.dev. (n)				
	R(calc.)	59.25			
	st.dev.(D6304-B:20)	69.181			
_	R(D6304-B:20)	193.71		range 30 – 2100 mg/kg	
Compar	re	00.00		00 07007 "	
	R(D6304-A:20)	88.96		range 20 – 25000 mg/kg	
	R(D6304-C:20)	69.77		range 20 – 360 mg/kg	
_					
<sup>450</sup> T	<del></del>		<del></del>		0.025
400 +					Kernel Density
					0.02
350 +					·······
300 +					
250 +					0.015 -
				_ Δ	
200		A A A	<u> </u>	<u> </u>	0.01
150	A A A A A A A		-		
100 - 4 4					
					0.005 -
50 -					
6 th	178 496 262 262 263 542 333	87.5 0002 325 235	986 179 179 182 183 194 196 196 196 196 196 196 196 196 196 196	9001 6004 933 963 360 435 435 435 437 437	0 100 200 200
1943	496 6359 614 614 614 6333 6333	181 32 32 623	1986 179 812 831 857 6283 1569 6016	901 1604 1023 349 360 360 1417 1417 1431	0 100 200 300
L					

# Determination of Water Separability at 54 °C, distilled water on sample #22077; results in minutes

	-	3 mL or less			•	complete break	test	time
	method	emulsion	z(targ)	37 mL of wate	r z(targ)	(40-40-0)	z(targ) aborted	
178								
179							No	30
225							Voc	20
237 325							Yes Yes	30 30
331	ISO6614	>60 f+?	>4.99	>60 f+?	>4.50	>60 f+?	>5.21 No	60
349	1000014							
360	ISO6614	23	-0.19				Yes	30
432								
451	D1401	20	-0.61	20	-1.10	20	-0.39	
496							Yes	30
542								
603	D4.404		0.40			 7		
614	D1401	6.8 C	-2.46	6.8	-2.94	7	-2.21 No	
633 657							Yes	30
663								
704								
840	D1401	>30		>30		>30	Yes	30
862								
901	D1401	>30		>30			Yes	30
912								
962								
963								
974								
1023 1059								
1146								30
1262	ISO6614	20	-0.61	> 60 f+?	>4.50	> 60 f+?	>5.21 Yes	60
1417	D1401	37	1.77	37	1.28	37	1.99 No	
1431								30
1435	D1401	41	2.33	41	1.84		No	
1569							Yes	30
1604								
1875 1943								
1943								
1966								
6002	ISO6614	19.8	-0.64	19.8	-1.12	19.8	-0.41 No	
6016							Yes	30
6141								
6235								
6283	D1401	27.2	0.40	27.2	-0.09	30	1.01 No	
6333				43	2.12		Yes	60
6359								
	normality	unknown		unknown		unknown		
	n	8		7		5		
	outliers	0		0		0		
	mean (n)	24.35		27.83		22.76		
	st.dev. (n)	10.782		13.267		11.401		
	R(calc.)	30.19 7.143		37.15 7.143		31.92 7.143		
	st.dev.(D1401:21)	7.143 20		7.143 20		7.143 20		
	R(D1401:21)	20		20		20		

Lab 614 first reported for time to reach 3 mL or less emulsion: 6 min 50 sec and for time to reach 37 mL or water: 6 min 50 sec



### Determination of Water Separability at 54 °C, distilled water on sample #22077; results in mL

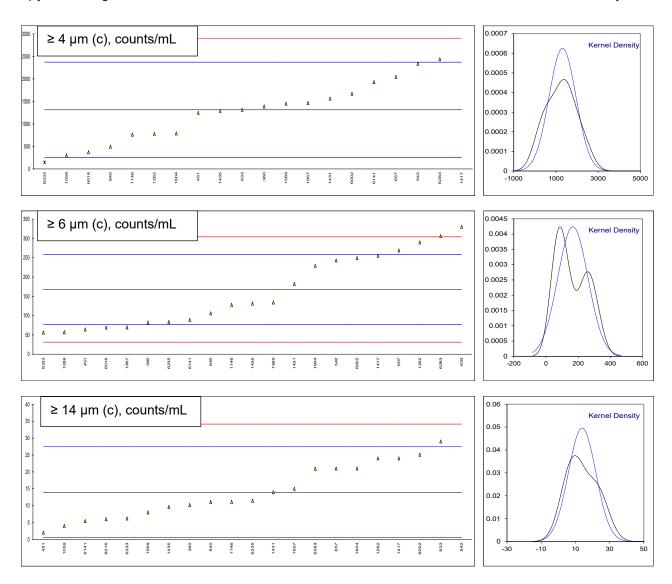
lab	method	volume oil phase	mark	volume water phase	mark	volume emulsion phase	mark
178							
	D1401	40		30		10	
225							
237	D1401	39	С	37	С	4	С
325		40		35		5	
	ISO6614	40		35		5	
349	1000011						
	ISO6614	42		35		3	
432	D4404						
451	D1401	40		40		0	
496	D1401	5		32		43	
542 603							
	D1401	40		40		0	
633	D1401						
	D1401	40	С	20		20	С
663	D1401		O				O
704							
840	D1401	41		35		4	
862							
901	D1401	42		33		5	
912							
962							
963							
974							
1023							
1059							
1146		43		30		7	
	ISO6614	41		36		3	
1417	D1401	40	_	40	0	0	
1431		40	С	37	С	3	
1435 1569	D1401	40		35		5	
1604	D1401	40				5	
1875							
1943							
1957							
1966							
	ISO6614	40		37		3	
6016	D1401	40	С	30	С	10	С
6141							
6235							
6283							
6333	D1401	42		38		0	
6359							

Lab 237 first reported for volume of oil phase: 36, for volume of water phase: 25 and for volume of emulsion phase: 19
Lab 657 first reported for volume of oil phase: 6 and for volume of emulsion phase: 65
Lab 1431 first reported for volume of oil phase: 45 and volume of water phase: 32
Lab 6016 first reported for volume of oil phase: 0, for volume of water phase 27 and for volume of emulsion phase: 53

### Determination of Level of Contamination on sample #22077; results in counts/mL

lab	method	≥ 4 µm (c)	) mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178										
179										
225										
237										
325										
331										
349										
360	ISO4406	1388.7		0.14	82.0		-1.88	10.2		-0.56
432										
451	ISO11500	1248		-0.12	64		-2.28	2		-1.78
496										
542	ISO11500	2341		1.94	243		1.66	109	R(0.01)	14.12
603									, ,	
614										
633	ISO4407	1315		0.00	330		3.57	29		2.24
657	ISO4406	2046		1.38	269		2.23	21		1.05
663										
704										
840	D7647	493.5		-1.55	105.9		-1.36	11.1		-0.42
862	2.0									
901										
912										
962										
963										
974										
1023										
	D7647			1.01			2.42	4		1 40
1059 1146	D7647	301 764.87		-1.91	57		-2.43 -0.88			-1.48 -0.42
1262	ISO11500	764.67 780		-1.04	127.60			11.13 24		-0.42 1.49
	130 1 1300		D(0.04)	-1.01	290		2.69			
1417	1004407	8122	R(0.01)	12.84	255		1.92	24		1.49
1431		1563		0.47	182		0.32	14		0.01
1435	ISO4407	1291.63		-0.04	131.13		-0.80	9.63		-0.64
1569	ISO11500	1449		0.26	134		-0.74	8		-0.89
1604	ISO4407	790		-0.99	229		1.35	21		1.05
1875										
1943	100440=							45.0		
1957	ISO4407	1465.3		0.29	69.3		-2.16	15.0		0.15
1966										
	D7647	1670.8		0.67	249.0		1.79	25.1		1.66
6016	D7596	371		-1.78	68	ex	<b>-</b> 2.19	6		-1.18
6141	ISO4406	1930.2		1.16	88.75		-1.73	5.42		-1.27
6235					83.44		-1.85	11.44		-0.37
	ISO4407	2438.73		2.12	306.13		3.04	20.93		1.04
6333	D7596	144.6	ex	-2.21	56.2		-2.45	6.2		-1.15
6359										
	normality	OK			OK			OK		
	n	18			20			20		
	outliers	1+1ex			0+1ex			1		
	mean (n)	1313.8			167.6			14.0		
	st.dev. (n)	638.29			94.11			8.05		
	R(calc.)	1787.2			263.5			22.5		
	st.dev.(D7647:10R18)				45.50			6.73		
	R(D7647:10R18)	1484.6			127.4			18.8		
	(= : : : : : : : : : : : : ;									

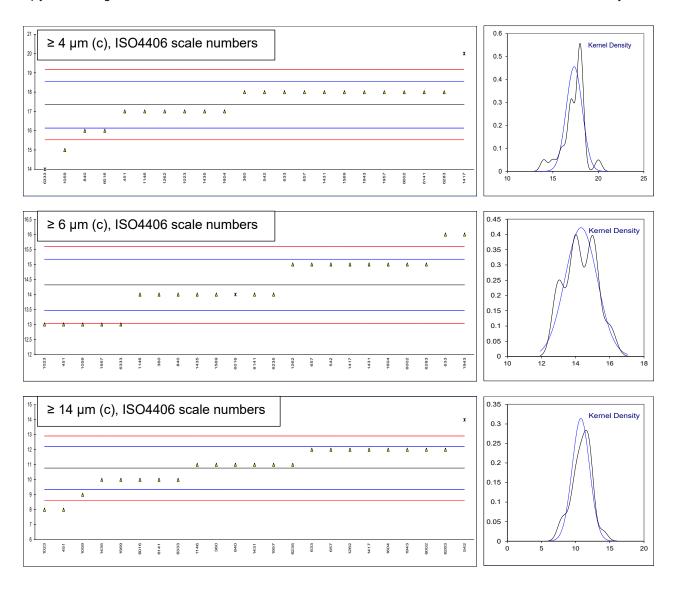
Lab 6016 test result excluded at  $\geq$  6  $\mu$ m as test result in counts/mL and ISO4406 scale number did not match Lab 6333 test result excluded at  $\geq$  4  $\mu$ m because of statistical outlier at related measurement in ISO4406 scale number



### Determination of Level of Contamination on sample #22077; results in ISO4406 scale numbers

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178										
179										
225										
237										
325										
331										
349										
360	ISO4406	18		1.07	14		-0.74	11		0.32
432										
451	ISO4406	17		-0.58	13		-3.08	8		-3.88
496										
542	ISO4406	18		1.07	15		1.59	14	ex	4.52
603	1001100								OX.	
614										
633	ISO4406	18		1.07	16		3.92	12		1.72
657	ISO4406	18		1.07	15		1.59	12		1.72
663	1004400									
704										
840	ISO4406	16		-2.22	14		-0.74	11		0.32
862	1004400			-2.22			-0.74			
901										
912										
962										
963										
974 1023	ISO4406	17		-0.58	13		2.00	8		-3.88
1023	ISO4406	15		-3.87	13		-3.08 -3.08	9		-3.66 -2.48
1146	ISO4406	17		-3.67 -0.58	14			11		0.32
1262	ISO4406	17		-0.58	15		-0.74 1.50	12		1.72
		20	0.4	4.36	15		1.59 1.59	12		
1417	ISO4406		ex							1.72
1431	ISO4406	18		1.07	15		1.59	11		0.32
1435	ISO4406	17		-0.58	14		-0.74	10		-1.08
1569	ISO11500	18		1.07	14		-0.74	10		-1.08
1604	ISO4406	17		-0.58	15		1.59	12		1.72
1875	1004400	40		4.07	40			40		4.70
1943	ISO4406	18		1.07	16		3.92	12		1.72
1957	ISO4406	18		1.07	13		-3.08	11		0.32
1966	D7047	40		4.07	4.5		4.50	40		4.70
6002	D7647	18		1.07	15		1.59	12		1.72
6016	ISO4406	16		-2.22	14	ex	-0.74	10		-1.08
6141	ISO4406	18		1.07	14		-0.74	10		-1.08
6235	1004400			4.07	14		-0.74	11		0.32
6283	ISO4406	18		1.07	15		1.59	12		1.72
6333		14	R(0.05)	-5.52	13		-3.08	10		-1.08
6359										
	normality	suspect			OK			OK		
	n	20			22			22		
	outliers	1+1ex			0+1ex			0+1ex		
	mean (n)	17.4			14.3			10.8		
	st.dev. (n)	0.88			0.95			1.27		
	R(calc.)	2.5			2.6			3.6		
	. ,	0.61			0.43			0.71		
	st.dev.(D7647:10R18)				1.2					
	R(D7647:10R18)	1.7			1.2			2.0		

Lab 542 test result excluded at  $\geq$ 14  $\mu$ m because of statistical outlier at related measurement in counts/mL Lab 1417 test result excluded at  $\geq$ 4  $\mu$ m because of statistical outlier at related measurement in counts/mL Lab 6016 test result excluded at  $\geq$ 6  $\mu$ m as test result in counts/mL and ISO4406 scale number did not match



#### **APPENDIX 2**

### Number of participants per country

- 1 lab in ARGENTINA
- 1 lab in AUSTRALIA
- 1 lab in AUSTRIA
- 2 labs in BELGIUM
- 2 labs in BULGARIA
- 1 lab in CHINA, People's Republic
- 1 lab in COTE D'IVOIRE
- 1 lab in FRANCE
- 1 lab in GEORGIA
- 2 labs in GERMANY
- 1 lab in GREECE
- 1 lab in INDIA
- 2 labs in ITALY
- 1 lab in KAZAKHSTAN
- 1 lab in LATVIA
- 2 labs in MALAYSIA
- 1 lab in NETHERLANDS
- 1 lab in NIGERIA
- 1 lab in NORWAY
- 1 lab in PHILIPPINES
- 3 labs in POLAND
- 3 labs in SAUDI ARABIA
- 1 lab in SINGAPORE
- 1 lab in SLOVENIA
- 3 labs in SPAIN
- 1 lab in THAILAND
- 1 lab in TURKEY
- 1 lab in UKRAINE
- 1 lab in UNITED ARAB EMIRATES
- 2 labs in UNITED KINGDOM
- 2 labs in UNITED STATES OF AMERICA
- 1 lab in VIETNAM

#### **APPENDIX 3**

#### **Abbreviations**

C = final test result after checking of first reported suspect test result

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ DG(0.05) &= \text{straggler in Double Grubbs' outlier test} \\ \end{array}$ 

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-? / false-? = possibly a false negative test result?

SDS = Safety Data Sheet

#### Literature

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