Results of Proficiency Test
Polyether-polyols
December 2006

Organised by: Institute for Interlaboratory Studies (i.i.s.)
Spijkenisse,the Netherlands

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CONTENTS

Appendices:

1 INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organise a proficiency test for the analysis of Polyether Polyol during the annual proficiency testing program 2006/2007. In this first international interlaboratory study 14 laboratories in 7 different countries have participated. See appendix 2 for a list of participants in alphabetical country order. In this report the results of the Polyether Polyol proficiency test are presented and discussed.

2 SET-UP

The Institute for Interlaboratory Studies (i.i.s.) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. In cooperation with 2 large producers was decided to send 2 different samples (1*500 mL of Diol (labelled 0689) and 1*500 mL of Triol (labelled 0690)). Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO guide 43 and ILAC-G13:2000. This ensures 100% confidentiality of participant's data. Also customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of November 2003 (i.i.s.-protocol, version 3.0).

2.3 SAMPLES

In this proficiency test two samples were used.
The necessary 10 litre bulk material for sample 0689 (Diol) was obtained from a local trader. After homogenisation, 20 subsamples were transferred to 500 mL brown glass bottles with inner and outer caps, labelled 0689. The homogeneity of the subsamples was checked by determination of Water in accordance with ASTM D4672:06e1 on 4 stratified random selected samples:

	Water in % wt
sample 0689-1	0.0940
sample 0689-2	0.0931
sample 0689-3	0.0928
sample 0689-4	0.0942

Table 1: homogeneity tests of subsamples 0689 (Diol)

From the results in table 1 the repeatabilities were calculated by multiplication of the standard deviations by 2.8:

	Water in % wt
r (observed)	0.0019
reference method	ASTM D 4672:06e1
r (reference method)	0.6903

Table 2: repeatability of subsamples 0689 (Diol)

The necessary 15 litre bulk material for the second sample 0690 (Triol) was also obtained from a local trader. After homogenisation, 24 subsamples were transferred to 500 mL brown glass bottles with inner and outer caps, labelled 0690. The homogeneity of the subsamples was checked by determination of Water in accordance with ASTM D4672:06e1 on 4 stratified random selected samples:

	Water in % wt
sample 0690-1	0.0171
sample 0690-2	0.0175
sample 0690-3	0.0178
sample 0690-4	0.0183

Table 3: homogeneity tests of subsamples 0690 (Triol)

From the results in table 3 the repeatabilities were calculated by multiplication of the standard deviations by 2.8:

	Water in % wt
r (observed)	0.0014
reference method	ASTM D 4672:06e1
r (reference method)	0.1305

Table 4: repeatability of subsamples 0690 (Triol)

The repeatabilities of the results of the homogeneity tests were in full agreement with the requirements as mentioned in the respective standards. Therefore, homogeneities of the both samples were assumed.

To each of the participating laboratories 2 bottles (1*500 mL of Diol labelled as 0689 and 1*500 mL of Triol labelled as 0690), were sent on December 21, 2006.

2.4 ANALYSES

The participants were asked to determine on sample 0689 (Diol): Acid number, Appearance, Ash, Colour Pt/Co, Hydroxyl Number, Kinematic Viscosity @ 25°C, Mol. weight calc. from OH%, pH (5% in H_2O), Residual EO and Water. On sample 0690 (Triol) the following determinations were requested: Acid Number, Appearance, Colour Pt/Co, Hydroxyl Number, Kinematic Viscosity @ 25°C, Potassium and Sodium, Unsaturates and Water.

To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also a letter of instructions and a MSDS were added to the package.

3 RESULTS

During four weeks after sample despatch the results of the individual laboratories were gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. Shortly after the deadline the available results were screened for suspect data. A 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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. After removal of outliers this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.
In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

Finally the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.
Statistical calculations were performed as described in the report ‘i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation, November 2003 (i.i.s.-protocol, version 3.0).

3.2 GRAPHICS

In order to visualise 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

results are plotted. The corresponding laboratory numbers are under the X-axis. A straight line presents the average of the reported data. Two striped lines present the reproducibility limits of the selected standard, calculated as mean ± target reproducibility, parallel to the average line. 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 (see appendix 3; nr.13 and 14).

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, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.
The target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated in accordance with:

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. 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 no problems were encountered with the despatch of the samples. All the participants did receive the samples and returned the test results before the final reporting date. The 14 reporting laboratories did send in 170 numerical results. Observed were 11 outlying results, which is 6.5 %. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

4.1 Evaluation per test

In this section the results are discussed per test.
Not all data sets proved to have a normal distribution. Not normal distribution was found in the determination of pH (5% in H_2O) on sample 0689 (Diol).
On sample 0690 (Triol) a not normal distribution was found in the determination of Unsaturates. Therefore, the statistical evaluation for these determinations should be used with care.

Sample 0689 (Diol):

Acid Number: This determination is not problematic. No results were outside the reproducibility limits. The calculated reproducibility is in good agreement with the requirement of ASTM D 4662:03.

Appearance: This determination is not problematic. Most of the laboratories agreed about the appearance of the sample 0689 (Diol) which is clear. The way of reporting varies strongly and could be improved.
Please note the results acc. to ASTM D 4670:02 are reported as presence or absence of suspended matter.

Ash: This determination is problematic. The reported results are between 0.003 – 0.094 %M/M. In this determination difference methods were used. Three results were outside the reproducibility limits. The calculated reproducibility is not at all in agreement with the requirements of ASTM D 1119:05, nor with the requirement of ASTM D 482:03 (R=0.005). Please note that ASTM D 2109 is not a suitable method for the determination of Ash.

Colour: This determination is problematic. Two results were outside the reproducibility limits. The calculated reproducibility is not in agreement with the requirement of ASTM D 1209:05. When the D 1209:05 data was evaluated separately, the calculated reproducibility is again not in agreement with the requirements of D1209:05.

Hydroxyl No.: This determination is problematic for one laboratory. One result was outside the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outlier, in agreement with the requirement of ASTM D 4274:05.

Kin. Visco.: No obvious analytical problems are observed. The reported results are between 81.1-84.1 cSt. Unfortunately, for this determination no precision data are available in ASTM D 445:06. Therefore no significant conclusions can be drawn. Rounding of results should be done with great care.

Mol. weight: Too few results were reported. Therefore, no meaningful conclusion could be drawn.

pH (5% in H_2O): This determination is problematic for at least one laboratory. The reported pH-values are between 5.25-7.3. Unfortunately, for this determination no precision data are available. Therefore no significant conclusions can be drawn.

Residual EO: Only one participant reported a result for this determination. Therefore no meaningful conclusions could be drawn.

Water: This determination is not problematic. No results were outside the reproducibility limits. The calculated reproducibility is in good agreement with the requirement of ASTM D 4672:06e1.

Sample 0690 (Triol):

Acid Number: This determination is not problematic. No results were outside the reproducibility limits. The calculated reproducibility is in full agreement with the requirement of ASTM D 4662:03.

Appearance: This determination is not problematic. All the laboratories agreed about the appearance of the sample 0689 (Triol) which is clear. The way of reporting varies strongly and could be improved.

Colour: This determination is problematic. Two results were outside the reproducibility limits. The calculated reproducibility is not in agreement with the requirement of ASTM D 1209:05. When the D 1209:05 data was evaluated separately, the calculated reproducibility is again not in agreement with the requirements of D1209:05.

Hydroxyl No.: This determination is somewhat problematic. No results were outside the reproducibility limits. The calculated reproducibility is not in agreement with the requirement of ASTM D 4274:05.

Kin. Visc: This determination is problematic for at least one laboratory. The reported results are between 554.1-662.4 cSt. Unfortunately, for this determination no precision data are available in ASTM D 445:06. Therefore no significant conclusions can be drawn. Rounding of results should be done with great care.

K & Na: This determination is problematic for at least one laboratory. All the reported results are between <1-2.1, except for one result. Unfortunately, for this determination no precision data are available. Therefore no significant conclusions can be drawn.

Unsaturates: This determination is not problematic. No results were outside the reproducibility limits. The calculated reproducibility is in good agreement with the requirement of ASTM D 4671:05. The data seems bimodally divided. When the both groups were evaluated separately, the calculated reproducibilities are still in agreement with the requirements of D 4671:05.

Water: No analytical problems are observed. No results were outside the reproducibility limits and the calculated reproducibility is in good agreement with the requirement of ASTM D 4672:06e1.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards), are compared in the next tables.

Parameters	unit	n	average	2.8 * sd	R(lit)
Acid Number	mg KOH/g	11	0.1772	0.0453	0.3000
Appearance		10	n.a.	n.a.	n.a.
Ash	% M/M	6	0.0397	0.0916	0.0079
Colour Pt/Co		8	7.7	13.3	7.0
Hydroxyl Number	mg KOH/g	10	277.095	8.069	9.411
Kinematic Viscosity @ 25°C	cSt	10	82.827	2.821	Unknown
Mol. weight calc. from OH%		4	248.296	546.759	Unknown
pH (5% in H_2O)		7	5.566	0.840	Unknown
Residual EO	mg/kg	1	n.a.	n.a.	n.a.
Water	% M/M	14	0.0966	0.0168	0.5117

Table 6: Reproducibilities of the tests results of sample 0689

Parameter	unit	n	Average	2.8 *sd_R	R (lit)
Acid Number	mg KOH/g	12	0.011	0.018	0.300
Appearance		10	n.a.	n.a.	n.a.
Colour Pt/Co		8	8.8	14.9	7.0
Hydroxyl Number	mg KOH/g	11	47.626	2.154	1.980
Kinematic Viscosity @ 25°C	cSt	9	560.014	16.115	Unknown
Potassium and Sodium	mg/kg	6	1.308	1.509	Unknown
Unsaturates	meq/g	9	0.0354	0.0330	0.0353
Water	% M/M	12	0.0152	0.0102	0.0803

Table 7: Reproducibilities of the tests results of sample 0690

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

4.3 THE PROFICIENCY TEST OF JANUARY 2007.

	January 2007
Number of reporting labs	14
Number of results reported	170
Statistical outliers	10
Percentage outliers	5.9 %

Table 8: Proficiency tests of January 2007

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

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

Determination	Sample 0689	Sample 0690
Acid Number	++	++
Appearance	++	n.a.
Ash	--	n.d.
Colour Pt/Co	--	--
Hydroxyl Number	+	--
Kinematic Viscosity @ 25°C	n.a.	n.a.
Mol. weight calc. from OH%	n.a.	n.d.
pH (5% in H_2O)	n.a.	n.d.
Residual EO	n.a.	n.d.
Water	++	++
Potassium and Sodium	n.d.	n.a.
Unsaturates	n.d.	+/-

Table 9: Comparison determinations against the standards

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

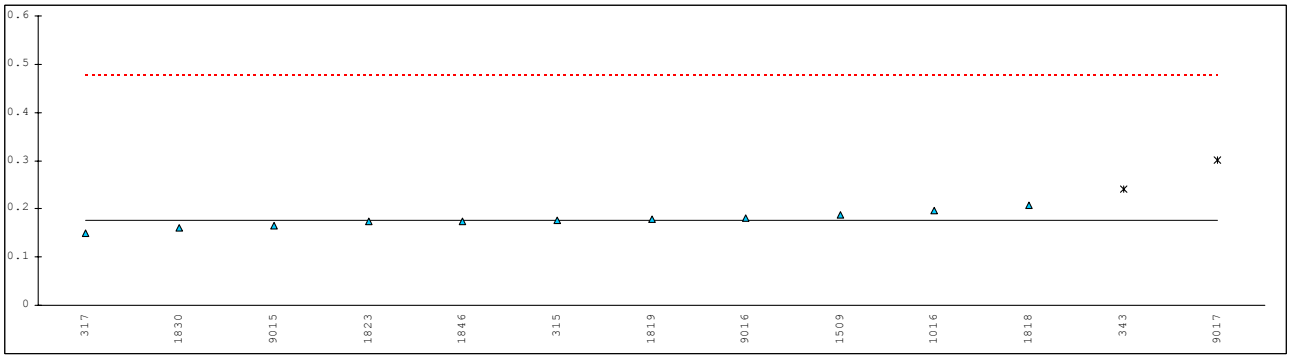
++:	group performed much better than the standard
+ :	group performed better than the standard
+/-:	group performance equals the standard
- :	group performed worse than the standard
-- :	group performed much worse than the standard
n.d.:	not determined
n.e.:	not evaluated
n.a.:	not applicable

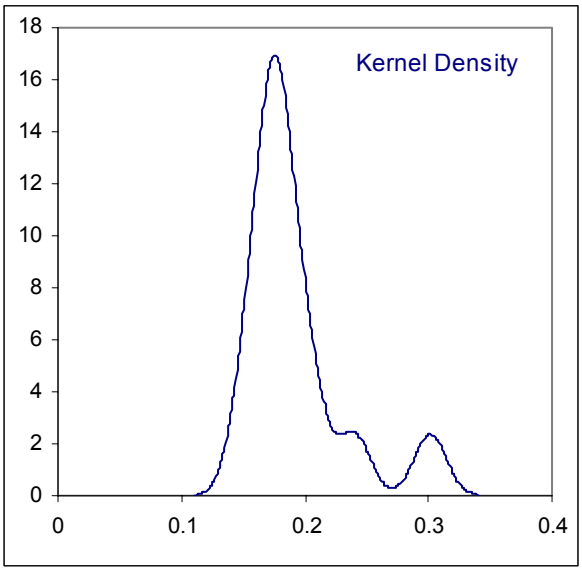
APPENDIX 1

Determination of Acid number on Diol sample 0689; results in mg KOH/g.

lab	method	value	mark	Z(targ)	remarks
315	D 4662	0.1760		-0.01	
317	INHOUSE	0.15		-0.25	
343	D 4462	0.240	C,G(0.05)	0.59	First reported 0.55
1016	D 4662	0.197		0.18	
1509	D 4662	0.188		0.10	
1818	INHOUSE	0.207		0.28	
1819	D 4662	0.179		0.02	
1823	D 4662	0.174		-0.03	
1830	INHOUSE	0.16		-0.16	
1846	INHOUSE	0.174		-0.03	
9003		-----		-----	
9015	D 4662	0.1650		-0.11	
9016	INHOUSE	0.17963		0.02	
9017	INHOUSE	0.3016	G(0.01)	1.16	

normality	OK
n	11
outliers	2
mean (n)	0.1772
st.dev. (n)	0.01616
R(calc.)	0.0453
R(D 4662:03)	0.3000





Determination of Appearance on Diol sample 0689;

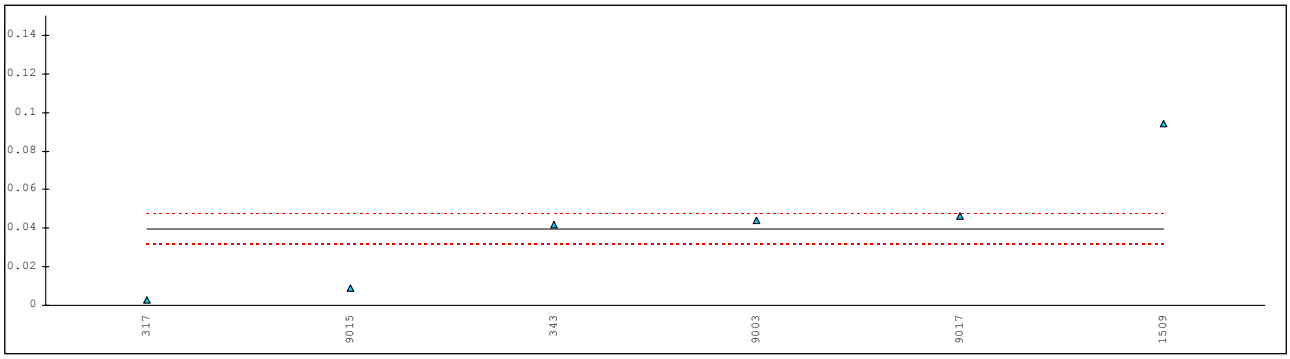
lab	method	value	mark	Z(targ)	remarks
315	INHOUSE	CFFSM		-----	
317	INHOUSE	B&C		-----	
343	D 4670	C&C		-----	
1016	INHOUSE	CLEAR/PASS		-----	
1509	INHOUSE	CFFSM		-----	
1818		-----		-----	
1819	D 4670	OK		-----	
1823	INHOUSE	CFFSM		-----	
1830		-----		-----	
1846		-----		-----	
9003	D 4670	CLEAR		-----	
9015	D 4670	PASS		-----	
9016		-----		-----	
9017	INHOUSE	CLEAR		-----	

normality	n.a.
n	10
outliers	0
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D 4670:02)	n.a.

B&C = Bright and Clear
C&C = Clear and Colourless
CFFSM = Clear and Free From Suspended Matter

Determination of Ash on Diol sample 0689; results in %M/M.

lab	method	value	mark	Z(targ)	remarks
315		-----		-----	
317	INHOUSE	0.003		-12.94	
343	INHOUSE	0.042		0.81	
1016		-----		-----	
1509	D 482	0.0942		19.21	
1818		-----		-----	
1819		-----		-----	
1823		-----		-----	
1830		-----		-----	
1846		-----		-----	
9003	INHOUSE	0.044		1.51	
9015	D 2109	0.0086803	U	-10.94	Reported in wrong unit, 86.803 ppm
9016		-----		-----	
9017	D 1119	0.0464		2.36	
	normality	OK			
	n	6			
	outliers	0			
	mean (n)	0.0397			
	st.dev. (n)	0.03270			
	R(calc.)	0.0916			
	R(D 1119:05)	0.0079			Compare R(D482:03) = 0.005

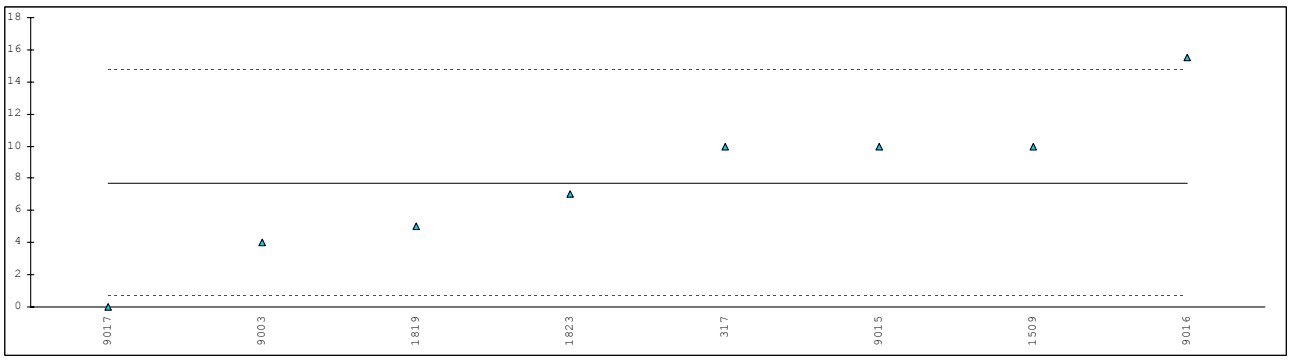


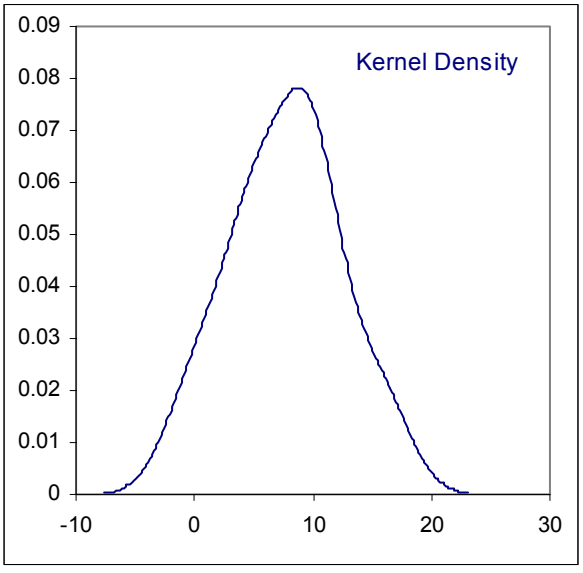
Determination of Colour Pt/Co on Diol sample 0689;

lab	method	value	mark	Z(targ)	remarks
315	D 1209	<5		<-1.08	
317	INHOUSE	10		0.92	
343	D 1209	<5		<-1.08	
1016		-----		-----	
1509	D 1209	10		0.92	
1818		-----		-----	
1819	D 1209	5		-1.08	
1823	D 1209	7		-0.28	
1830		-----		-----	
1846		-----		-----	
9003	D 1209	4.0		-1.48	
9015	D 1209	10		0.92	
9016	D 4890	15.51		3.13	
9017	D 4890	0		-3.08	

		All values (*):	Only D 1209 data (*):
normality	OK	OK	OK
n	8	10	7
outliers	0	0	0
mean (n)	7.7	6.7	5.9
st.dev. (n)	4.75	4.72	3.22
R(calc.)	13.3	13.2	9.0
R(D 1209:05)	7.0	7.0	7.0

*) In the calculation of the mean, standard deviation, the reproducibility and below graph, a reported value of <x is changed in x/2 (for example <5 into 2.5).

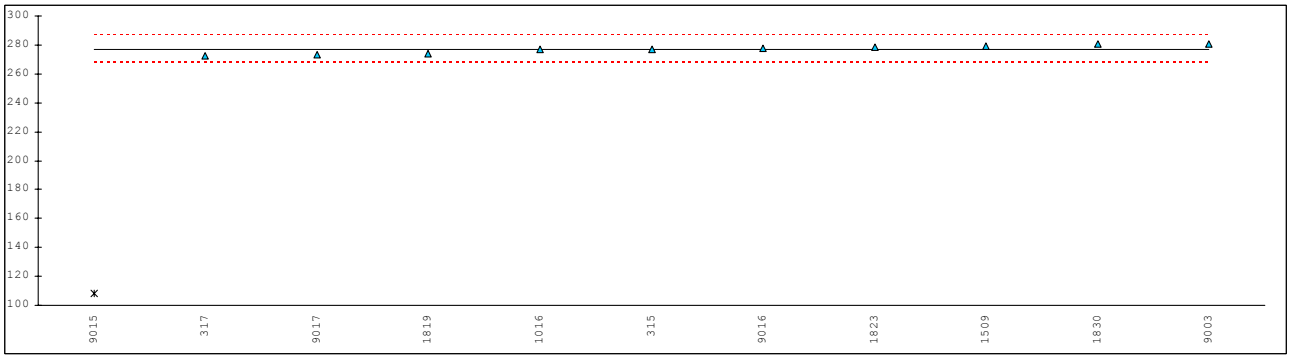


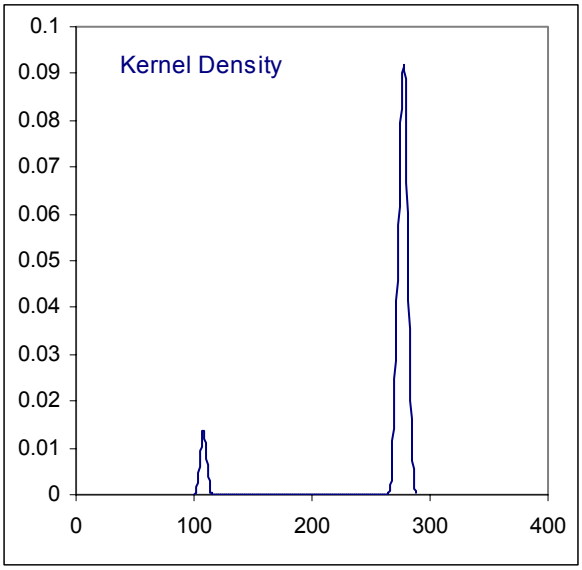


Determination of Hydroxyl Number on Diol sample 0689; results in mg KOH/g.

lab	method	value	mark	Z(targ)	remarks
315	D 4274	277.25		0.05	
317	INHOUSE	272.7		-1.31	
343		-----		-----	
1016	D 4274	277.2		0.03	
1509	D 4274	279.32		0.66	
1818		-----		-----	
1819	D 4274	274.34		-0.82	
1823	D 4274	278.63		0.46	
1830	INHOUSE	280.35		0.97	
1846		-----		-----	
9003	INHOUSE	280.7		1.07	
9015	D 4274-D	108.0628	G(0.01)	-50.29	
9016	D 4274	277.46	C	0.11	First reported 8.408
9017	D 4274-D	273		-1.22	

normality	OK
n	10
outliers	1
mean (n)	277.095
st.dev. (n)	2.8818
R(calc.)	8.069
R(D 4274:05)	9.411

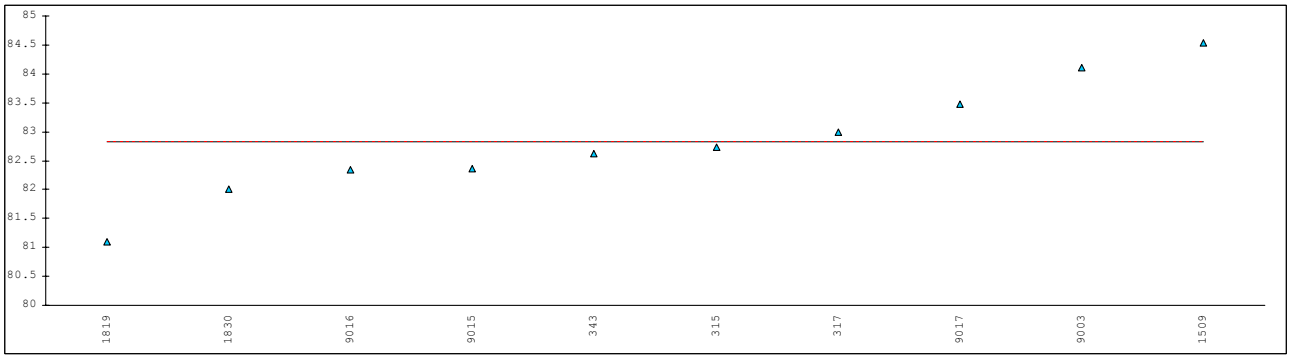


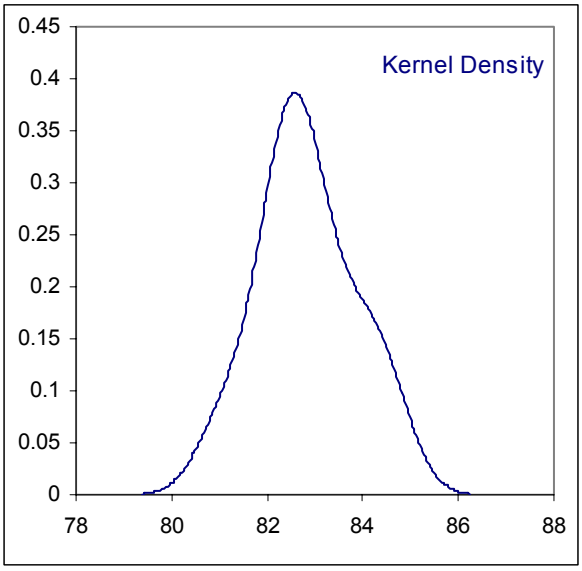


Determination of Kinematic Viscosity on Diol sample 0689; results in cSt.

lab	method	value	mark	Z(targ)	remarks
315	D 445	82.735		-----	
317	INHOUSE	82.99		-----	
343	D 445	82.624		-----	
1016		-----		-----	
1509	D 445	84.530		-----	
1818		-----		-----	
1819	D 445	81.1		-----	
1823		-----		-----	
1830	INHOUSE	82		-----	
1846		-----		-----	
9003	D 445	84.1		-----	
9015	D 445	82.3675		-----	
9016	D 445	82.34		-----	
9017	D 445	83.48		-----	

normality	OK
n	10
outliers	0
mean (n)	82.827
st.dev. (n)	1.0076
R(calc.)	2.821
R(D 445:06)	Unknown

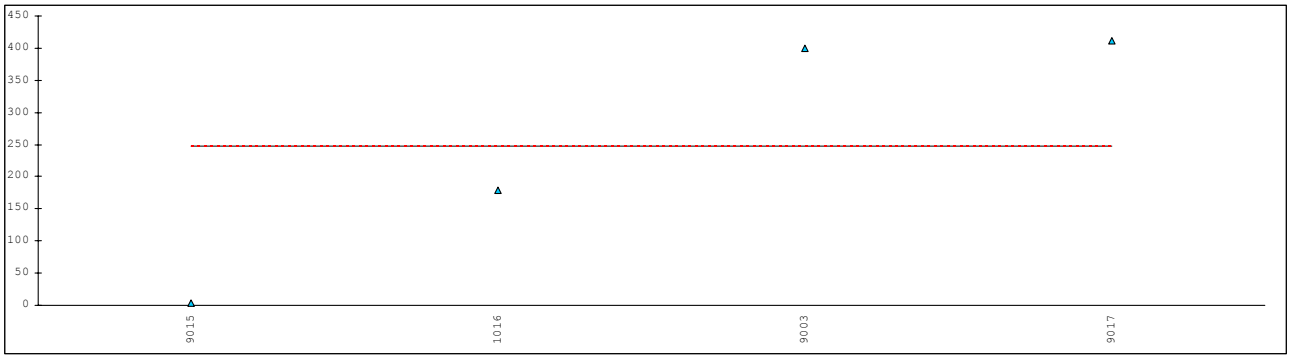




Determination of Mol. weight calc. from OH% on Diol sample 0689;

lab	method	value	mark	Z(targ)	remarks
315		-----		-----	
317		-----		-----	
343		-----		-----	
1016	INHOUSE	178.79		-----	
1509		-----		-----	
1818		-----		-----	
1819		-----		-----	
1823		-----		-----	
1830		-----		-----	
1846		-----		-----	
9003	INHOUSE	400		-----	
9015	D 4174-D	3.27463		-----	
9016		-----		-----	
9017	INHOUSE	411.12		-----	

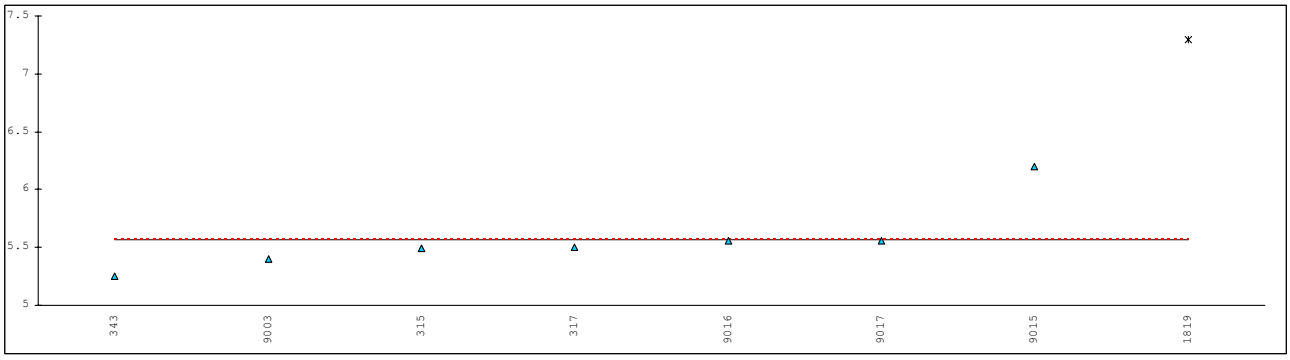
normality	OK
n	4
outliers	0
mean (n)	248.296
st.dev. (n)	195.2710
R(calc.)	546.759
R(lit)	Unknown

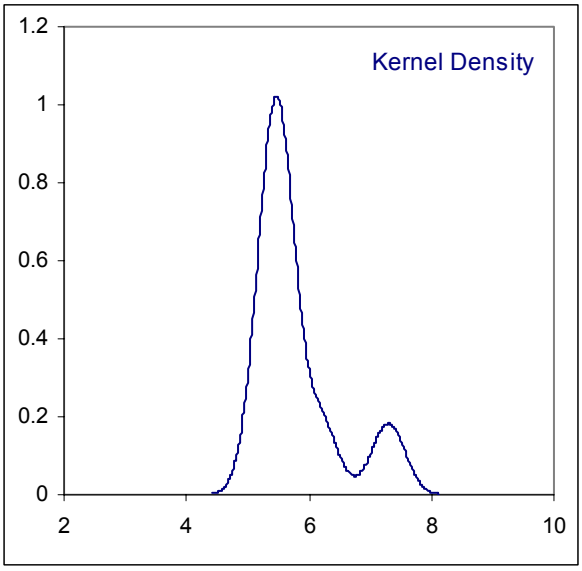


Determination of pH (5% in H_2O) on Diol sample 0689;

lab	method	value	mark	Z(targ)	remarks
315	INHOUSE	5.49		-----	
317	INHOUSE	5.5		-----	
343	INHOUSE	5.25		-----	
1016		-----		-----	
1509		-----		-----	
1818		-----		-----	
1819	INHOUSE	7.3	G(0.05)	-----	
1823		-----		-----	
1830		-----		-----	
1846		-----		-----	
9003	INHOUSE	5.4		-----	
9015	INHOUSE	6.20		-----	
9016	INHOUSE	5.56		-----	
9017	D 1209	5.56		-----	

normality	not OK
n	7
outliers	1
mean (n)	5.566
st.dev. (n)	0.2999
R(calc.)	0.840
R(lit)	Unknown





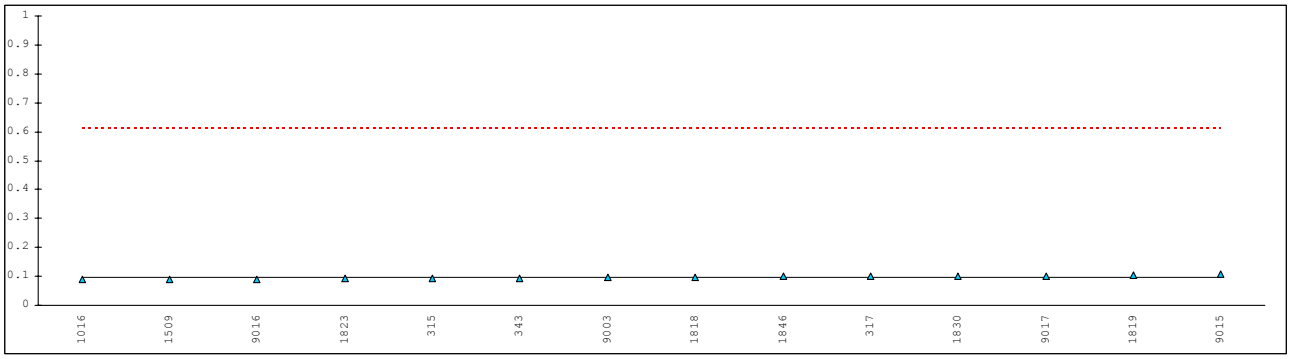
Determination of Residual EO on Diol sample 0689; results in mg/kg.

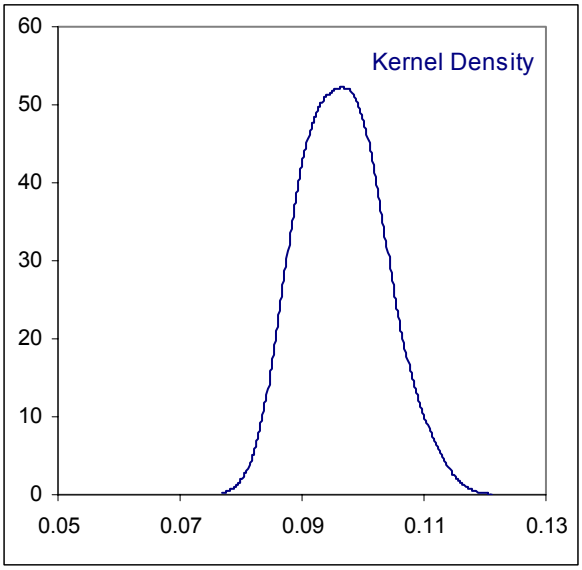
lab	method	value	mark	Z(targ)	remarks
315		-----		-----	
317		-----		-----	
343		-----		-----	
1016		-----		-----	
1509		-----		-----	
1818		-----		-----	
1819		-----		-----	
1823		-----		-----	
1830		-----		-----	
1846		-----		-----	
9003	INHOUSE	1.2		-----	
9015		-----		-----	
9016		-----		-----	
9017		-----		-----	
	normality	n.a.			
	n	1			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of Water on Diol sample 0689; results in %M/M.

lab	method	value	mark	Z(targ)	remarks
315	E 203	0.0929		-0.02	
317	INHOUSE	0.10		0.02	
343	ISO12937	0.0937	C	-0.02	First reported 0.0152
1016	D 4672	0.089		-0.04	
1509	D 4672	0.089		-0.04	
1818	D 4672	0.09713		0.00	
1819	E 203	0.103		0.04	
1823	D 4672	0.092		-0.02	
1830	INHOUSE	0.10		0.02	
1846	D 4672	0.099		0.01	
9003	E 203	0.096		0.00	
9015	E 203	0.10902		0.07	
9016	E 203	0.089		-0.04	
9017	E 203	0.102		0.03	

normality	OK
n	14
outliers	0
mean (n)	0.0966
st.dev. (n)	0.00602
R(calc.)	0.0168
R(D 4672:06e1)	0.5117

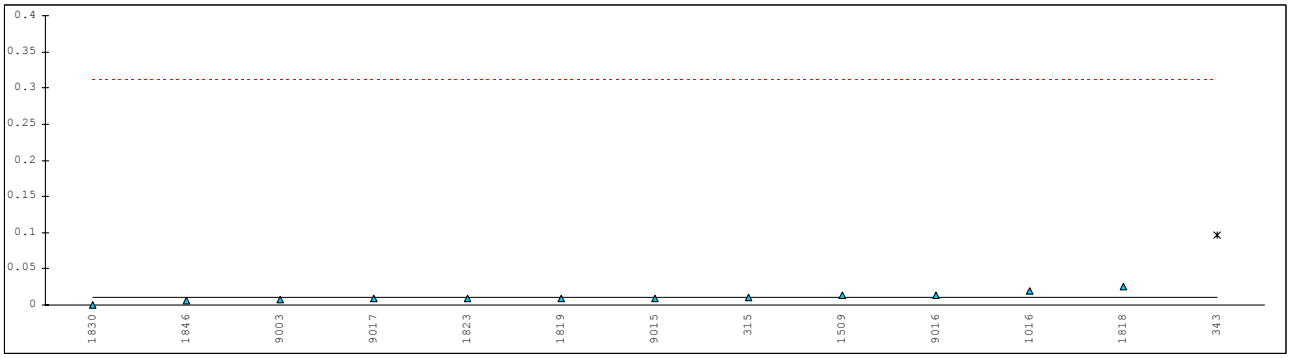


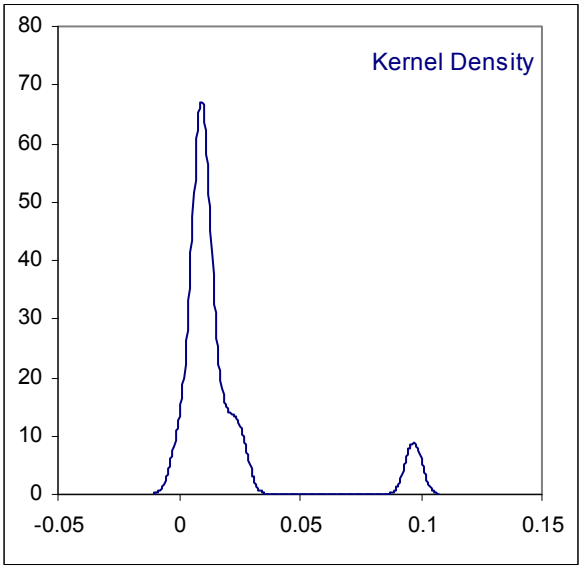


Determination of Acid Number on Triol sample 0690; results in mg KOH/g.

lab	method	value	mark	Z(targ)	remarks
315	D 4662	0.00977		-0.01	
317	INHOUSE	<0.01		-----	
343	D 4662	0.097	C,G(0.01)	0.80	First reported 0.414
1016	D 4662	0.020		0.08	
1509	D 4662	0.013		0.02	
1818	INHOUSE	0.0255		0.14	
1819	D 4662	0.009		-0.02	
1823	D 4662	0.009		-0.02	
1830	INHOUSE	0.00		-0.10	
1846	INHOUSE	0.006		-0.05	
9003	INHOUSE	0.008		-0.03	
9015	D 4662	0.0093		-0.02	
9016	INHOUSE	0.01374		0.03	
9017	INHOUSE	0.0085		-0.02	

normality	OK
n	12
outliers	1
mean (n)	0.011
st.dev. (n)	0.0066
R(calc.)	0.018
R(D 4662:03)	0.300





Determination of Appearance on Triol sample 0690;

lab	method	value	mark	Z(targ)	Remarks
315	INHOUSE	BCFFSM		-----	
317	INHOUSE	B&C		-----	
343	D 4670	C&C		-----	
1016	INHOUSE	CLEAR/PASS		-----	
1509	INHOUSE	CFFSM		-----	
1818		-----		-----	
1819	D 4670	OK		-----	
1823	INHOUSE	CFFSM		-----	
1830		-----		-----	
1846		-----		-----	
9003	D 4670	CLEAR		-----	
9015	D 4670	PASS		-----	
9016		-----		-----	
9017	INHOUSE	CLEAR		-----	

normality	n.a.
n	10
outliers	0
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D 4670:02)	n.a.

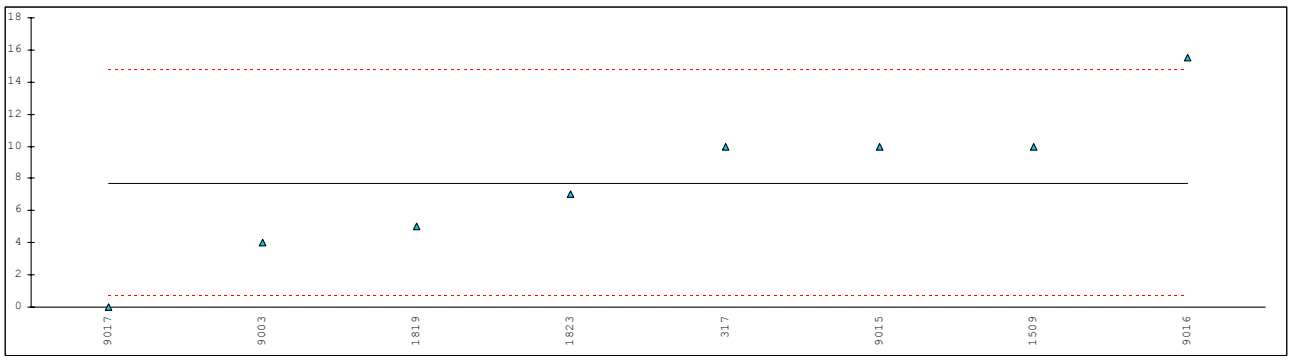
B&C = Bright and Clear
C&C = Clear and Colourless
BCFFSM = Bright, Clear and Free From Suspended Matter
CLFSM = Clear Liquid Free of Suspended Matter
CFFSM = Clear and Free From Suspended Matter

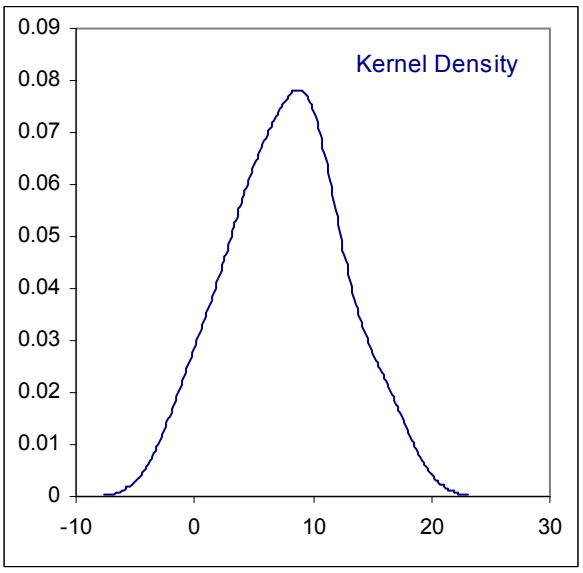
Determination of Colour Pt/Co on Triol sample 0690;

lab	method	value	mark	Z(targ)	remarks
315	D 1209	<10		<0.48	
317	INHOUSE	11		0.87	
343	D 1209	<5		<-1.52	
1016		-----		-----	
1509	D 1209	10		0.47	
1818		-----		-----	
1819	D 1209	5		-1.53	
1823	D 1209	6		-1.13	
1830		-----		-----	
1846		-----		-----	
9003	D 1209	7.6		-0.49	
9015	D 1209	10		0.47	
9016	D 4890	19.49		4.27	
9017	D 4890	1.5		-2.93	

		All results (*):	Only D 1209 data (*):
normality	OK	OK	OK
n	8	10	7
outliers	0	0	0
mean (n)	8.8	7.8	6.6
st.dev. (n)	5.33	5.20	2.78
R(calc.)	14.9	14.6	7.8
R(D 1209:05)	7.0	7.0	7.0

*) In the calculation of the mean, standard deviation, the reproducibility and below graph, a reported value of x< is changed in x/2 (for example <10 into 5).

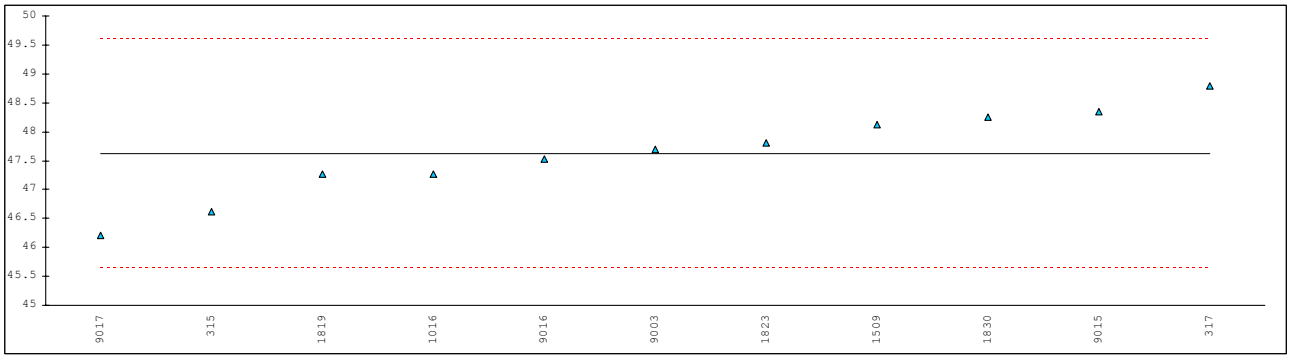


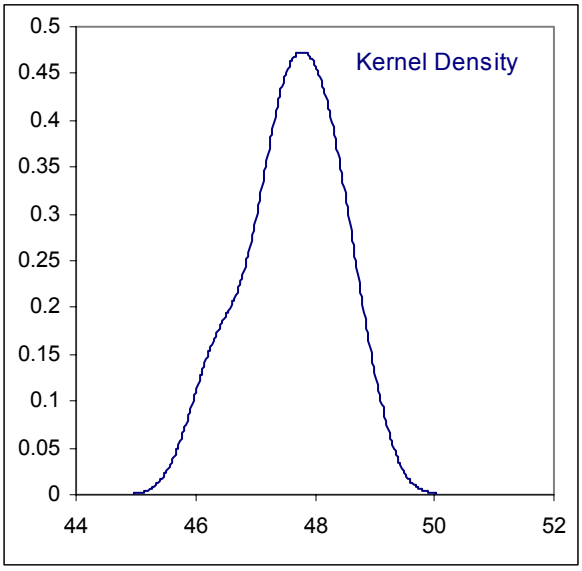


Determination of Hydroxyl Number on Triol sample 0690; results in mg KOH/g.

lab	method	value	mark	Z(targ)	remarks
315	D 4274	46.614		-1.43	
317	INHOUSE	48.8		1.66	
343		-----		-----	
1016	D 4274	47.26		-0.52	
1509	D 4274	48.13		0.71	
1818		-----		-----	
1819	D 4274	47.26		-0.52	
1823	D 4274	47.81		0.26	
1830	INHOUSE	48.25		0.88	
1846		-----		-----	
9003	INHOUSE	47.7		0.10	
9015	D 4274-D	48.34647		1.02	
9016	D 4274	47.52		-0.15	
9017	D 4274-D	46.2		-2.02	

normality	OK
n	11
outliers	0
mean (n)	47.626
st.dev. (n)	0.7692
R(calc.)	2.154
R(D 4274:05)	1.980

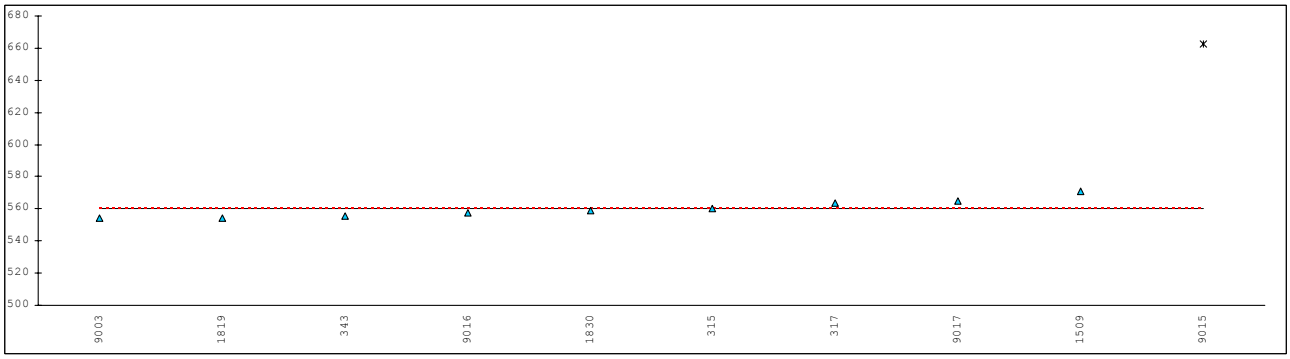


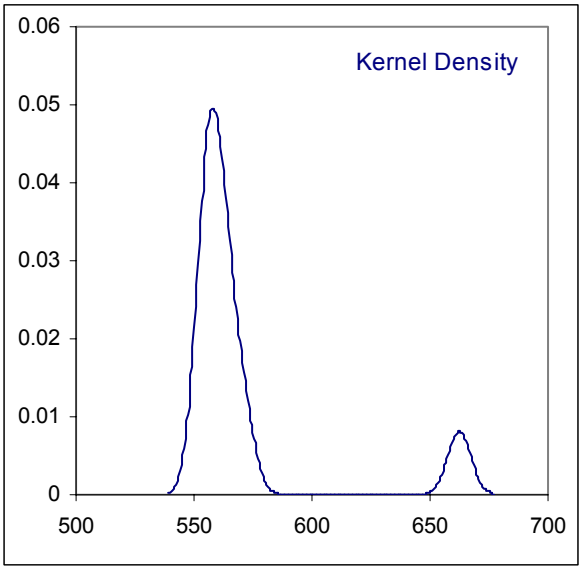


Determination of Kinematic Viscosity @ 25°C on Triol sample 0690; results in cSt.

lab	method	value	mark	Z(targ)	remarks
315	D 445	560.05		-----	
317	D 445	563.5		-----	
343	D 445	555.48		-----	
1016		-----		-----	
1509	D 445	571.26		-----	
1818		-----		-----	
1819	D 445	554.1		-----	
1823		-----		-----	
1830	INHOUSE	559		-----	
1846		-----		-----	
9003	D 445	554		-----	
9015	D 445	662.43513	G(0.01)	-----	
9016	D 445	557.5		-----	
9017	D 445	565.24		-----	

normality	OK
n	9
outliers	1
mean (n)	560.014
st.dev. (n)	5.7553
R(calc.)	16.115
R(D 445:06)	Unknown

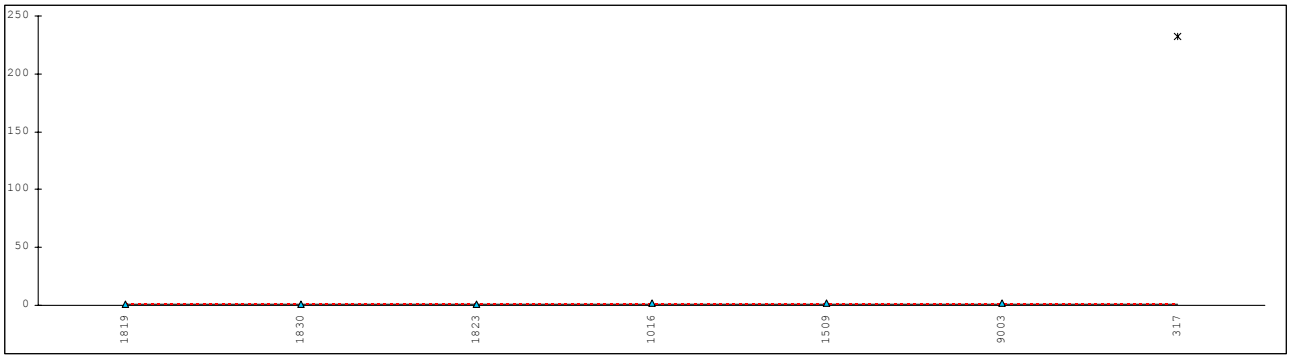


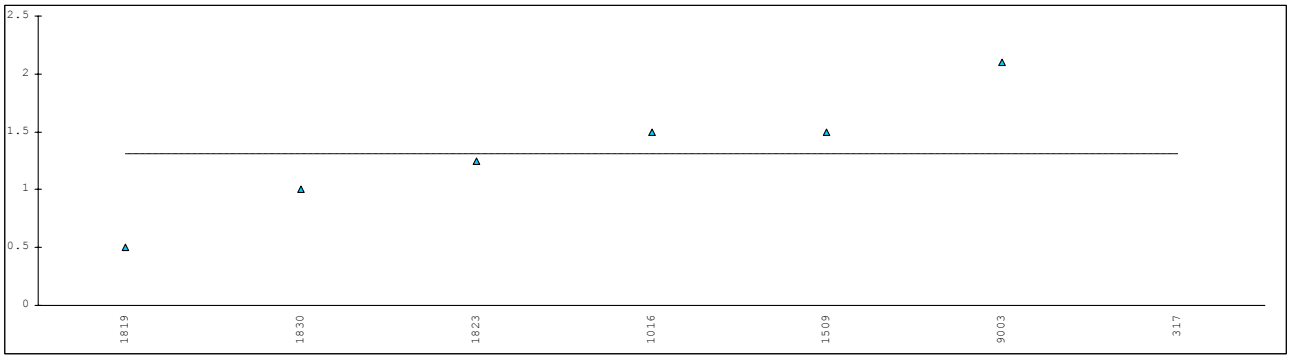


Determination of Potassium and Sodium on Triol sample 0690; results in mg/kg.

lab	method	value	mark	Z(targ)	remarks
315		-----		-----	
317	INHOUSE	232	G(0.01)	-----	
343	INHOUSE	<1		-----	
1016	NEN6426	1.5		-----	
1509	INHOUSE	1.50		-----	
1818		-----		-----	
1819	INHOUSE	0.5		-----	
1823	INHOUSE	1.25		-----	
1830	INHOUSE	1		-----	
1846		-----		-----	
9003	INHOUSE	2.1		-----	
9015		-----		-----	
9016		-----		-----	
9017		-----		-----	

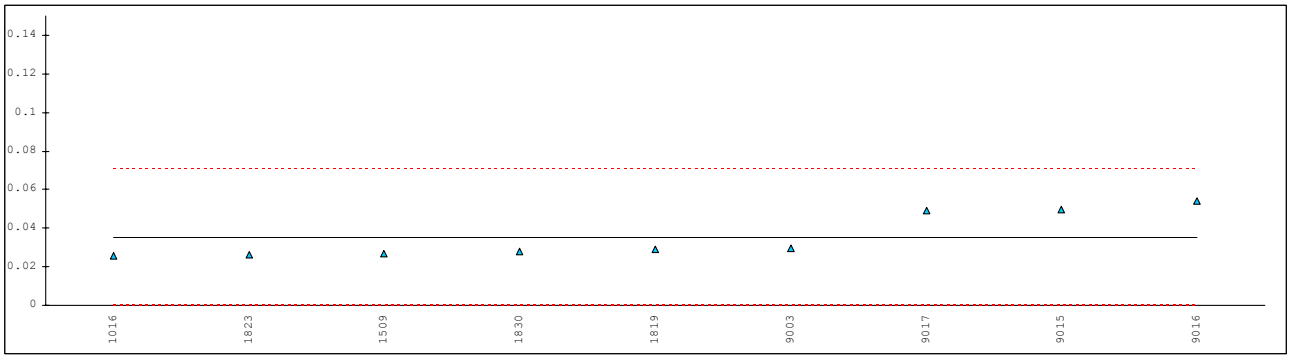
normality	OK
n	6
outliers	1
mean (n)	1.308
st.dev. (n)	0.5389
R(calc.)	1.509
R(lit)	Unknown

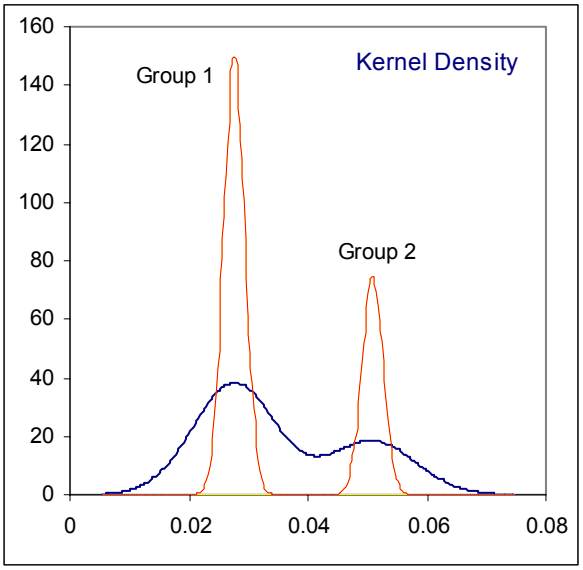




Determination of Unsaturates on Triol sample 0690; results in meq/g.

lab	method	value	mark	Z(targ)	remarks	
315		-----		-----		
317		-----		-----		
343		-----		-----		
1016	D 4671	0.0257	C	-0.77	First reported 0.013	
1509	D 4671	0.0270		-0.66		
1818		-----		-----		
1819	D 4671	0.029		-0.50		
1823	D 4671	0.0264		-0.71		
1830	INHOUSE	0.028		-0.58		
1846		-----		-----		
9003	INHOUSE	0.0296		-0.46		
9015	D 4671	0.0495		1.12		
9016	D 4671	0.0542		1.49		
9017	INHOUSE	0.0489		1.07		
					Only group 1data:	Only group 2 data:
	normality	not OK			OK	not OK
	n	9			6	3
	outliers	0			0	0
	mean (n)	0.0354			0.0276	0.0509
	st.dev. (n)	0.01178			0.00152	0.00290
	R(calc.)	0.0330			0.0043	0.0081
	R(D 4671:05)	0.0353			0.0046	0.0087

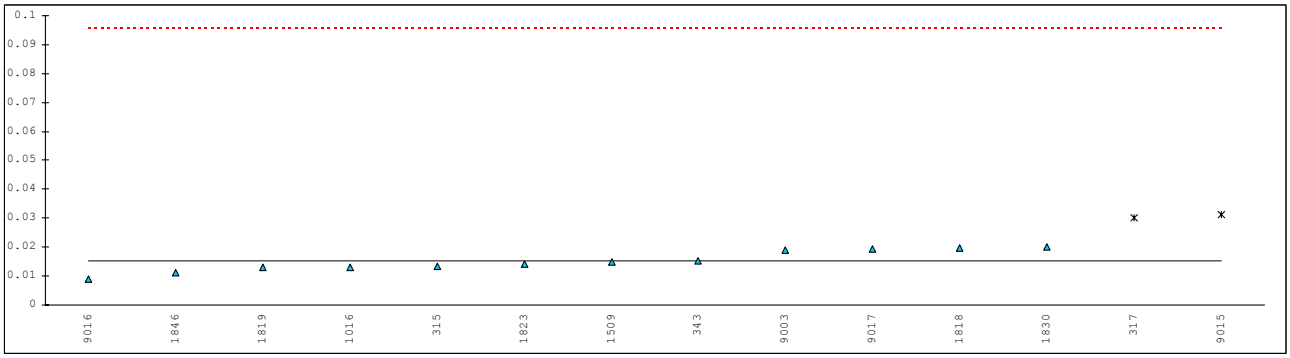


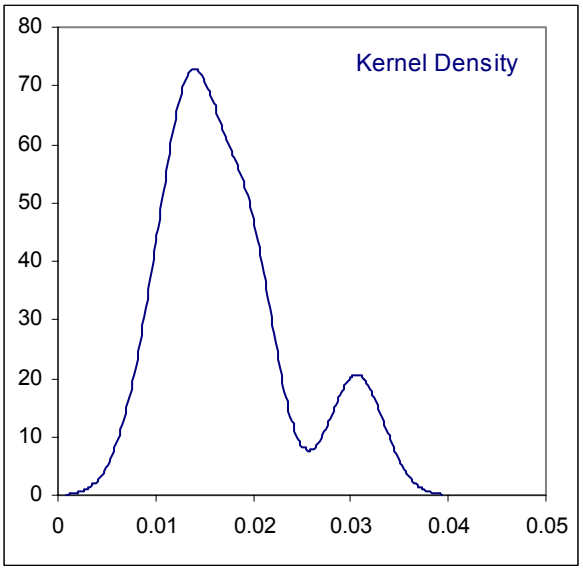


Determination of Water on Triol sample 0690; results in %M/M.

lab	method	value	mark	Z(targ)	remarks
315	E203	0.0135		-0.06	
317	INHOUSE	0.03	DG(0.05)	0.52	
343	ISO12937	0.0152	C	0.00	First reported 0.0937
1016	D4672	0.013		-0.07	
1509	D4672	0.015		-0.01	
1818	D4672	0.0198		0.16	
1819	E203	0.013		-0.07	
1823	D4672	0.014		-0.04	
1830	INHOUSE	0.02		0.17	
1846	D4672	0.011		-0.14	
9003	E203	0.019		0.13	
9015	E203	0.03121	DG(0.05)	0.56	
9016	E203	0.009		-0.21	
9017	E203	0.0193		0.14	

normality	OK
n	12
outliers	2
mean (n)	0.0152
st.dev. (n)	0.00364
R(calc.)	0.0102
R(D4672:06e1)	0.0803





APPENDIX 2

List of participants

Number of laboratories	Country
1 laboratory in	BELGIUM
1 laboratory in	BRAZIL
1 laboratory in	P.R.of CHINA
1 laboratory in	SINGAPORE
3 laboratories in	SPAIN
6 laboratories in	THE NETHERLANDS
1 laboratory in	U.S.A.

APPENDIX 3

Abbreviations:

C	= corrected result after checking of first reported suspect 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
EX	= excluded from calculations
S	= scope of the reported method is not applicable
U	= reported in wrong unit
W	= withdrawn result on request of participant
MSDS	= Material Safety Data Sheet

Literature:

1 i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation, November 2003 (i.i.s.-protocol, version 3.0).

2 W. Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)

3 ASTM E178-89

4 ASTM E1301-89

5 ISO 5725-86

6 ISO 5725, parts 1-6, 1994

7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)

8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)

9 IP 367/84

10 DIN 38402 T41/42

11 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)

12 J.N. Miller, Analyst, 118, 455, (1993)

13 Analytical Methods Committee Technical brief, No 4.January 2001

14 The Royal Society of Chemistry 2002, Analyst, 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)