Results of Proficiency Test Dissolved Gas Analysis November 2018

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1 INTRODUCTION

Since 2007 the Institute for Interlaboratory Studies organizes a proficiency test for the analyses on Dissolved Gas Analysis (DGA) in Transformer Oil every year. During the annual proficiency testing program 2018/2019, it was decided to continue the round robin for the analyses on Dissolved Gas Analysis (DGA) in Transformer Oil. In this interlaboratory study on DGA in Transformer Oil, 60 laboratories from 31 different countries did register for participation. See appendix 3 for the number of participants per country. In this report the results of the 2018 interlaboratory study on Dissolved Gas Analysis (DGA) in Transformer Oil 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 organiser of this proficiency test (PT). In total one batch of 65 certified syringes was prepared with lot code RN334. The syringes were prepared in two different volumes: 50mL and 100mL. The syringes (True North) were provided by Morgan Schaffer Inc, Quebec, Canada. Each syringe was uniquely numbered and labelled #18234. It was decided to send to each participating laboratory one syringe (50 or 100 mL) without the Morgan Schaffer certificate. 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/IEC 17043:2010 (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.

Morgan Schaffer Inc. is ISO 9001 certified and ISO/IEC17025 accredited by ANSI-ASQ.

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 Organization, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol can be downloaded from 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

In this proficiency test one type of Transformer Oil was used for the preparation of the gas tight syringes of 50mL or 100mL. These syringes were subsequently tested by Morgan Schaffer Inc. (Quebec, Canada) in accordance with ASTM Method D3612 and IEC 60567. In total one batch of 65 syringes (30x50mL and 35x100mL) was prepared (lot code RN334). Each syringe was labelled #18234.

The repeatabilities are in agreement for all components with 0.3 times the corresponding reproducibility of the target test method according with the procedure of ISO13528. Therefore, homogeneity of the subsamples #18234 was assumed.

Depending on the registration to each of the participating laboratories a syringe of 50mL or a syringe of 100mL, both labelled #18234, was sent on October 31, 2018. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

Morgan Schaffer declares that bulk storage prior to shipping has a shelf life of at least 6 months. This was assumed to be sufficient for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on sample #18234: Hydrogen, Oxygen, Nitrogen, Carbon Monoxide, Carbon Dioxide, Methane, Ethane, Ethane, Ethyn, Propane and Propene. Also, some analytical details were requested.

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 appropriate reference test methods 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 reanalysis). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the test 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 organisation 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.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

Finally, the reproducibilities were calculated from the standard deviations by multiplying these 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 analysis 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. The Kernel Density Graph 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 was projected over the Kernel Density Graph for reference.

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 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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:

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

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

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

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

4 EVALUATION

In this proficiency test, no major problems were encountered with the dispatch of the samples. Seven participants did not report any test results at all. Not all participants were able to report all requested components. Finally, 53 participants reported 496 numerical test results. Observed were 25 outlying test results, which is 5.0% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original 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 COMPONENT

In this section the reported test results are discussed per component. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 4.

Sample #18234

<u>Hydrogen:</u> The determination of this component was problematic. Three statistical

outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the strict requirements of

IEC 60567:2011.

Oxygen: The determination of this component was problematic for a number of

laboratories. Six statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in

agreement with the requirements of IEC 60567:2011.

Nitrogen: The determination of this component was problematic. Two statistical

outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of IEC

60567:2011.

<u>Carbon Monoxide:</u> The determination of this component was problematic. One statistical

outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of IEC

60567:2011.

<u>Carbon Dioxide</u>: The determination of this component was problematic. Five statistical

outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of IEC

60567:2011.

Methane: The determination of this component was not problematic. One

statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements

of IEC 60567:2011.

Ethane: The determination of this component was problematic. Two statistical

outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of IEC

60567:2011.

Ethene: The determination of this component was not problematic. Two

statistical outliers were observed. However, the calculated

reproducibility after rejection of the statistical outliers is in agreement

with the requirements of IEC 60567:2011.

Ethyn: The determination of this component was problematic. Two statistical

outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of IEC

60567:2011.

<u>Propane:</u> The determination of this component was problematic. The test results

of five participants were excluded. These participants detected Propane. After consulting Morgan Schaffer it became clear that

Propane was not present in the sample. Ten other participants agreed on a Propane level <10 μ l/L. Therefore, no z-scores were calculated.

<u>Propene:</u> The determination of this component was problematic. One statistical

outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of IEC

60567:2011.

4.2 Performance evaluation for the group of Laboratories

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * sd) and the target reproducibilities derived from reference test method (in casu IEC60567 test method) are compared in next table.

Component	unit	n	average	2.8 * sd	R(lit)
Hydrogen H ₂	μl/L	50	533	147	107
Oxygen O ₂	μl/L	46	15499	3358	3100
Nitrogen N ₂	μl/L	50	56117	18387	11223
Carbon Monoxide CO	μl/L	52	512	141	102
Carbon Dioxide CO ₂	μl/L	48	555	143	111
Methane CH₄	μl/L	52	518	111	104
Ethane C ₂ H ₆	μl/L	51	536	138	107
Ethene C ₂ H ₄	μl/L	50	524	114	105
Ethyn C ₂ H ₂	μl/L	50	510	127	102
Propane C₃H ₈	μl/L	10	<10	n.a.	n.a.
Propene C ₃ H ₆	μl/L	9	562	194	112

Table 1: reproducibilities of components on sample #18234

Without further statistical calculations it can be concluded from the overview given in table 1 that for most determinations it is difficult to get a compliance of the performance of the group of participating laboratories with the relevant standard IEC 60567:2011.

The problematic components have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2018 WITH PREVIOUS PTS

	November 2018	November 2017	November 2016	November 2015	November 2014
Number of reporting labs	53	61	53	45	40
Number of test results reported	496	580	487	401	358
Number of statistical outliers	25	21	57	29	10
Percentage of statistical outliers	5.0%	3.6%	11.7%	7.2%	2.8%

Table 2: comparison of statistical summary parameters with previous proficiency tests

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

The performance of the determinations of the proficiency tests was compared, expressed as relative standard deviation (RSD) of the PTs, see below table.

Determination	November 2018	November 2017	November 2016	November 2015	November 2014	IEC605671
Hydrogen H ₂	10%	15%	10%	21%	24%	7%
Oxygen O ₂	8%	12%	13%	19%	18%	7%
Nitrogen N ₂	12%	19%	13%	16%	13%	7%
Carbon Monoxide CO	10%	16%	12%	11%	12%	7%
Carbon Dioxide CO ₂	9%	15%	16%	12%	17%	7%
Methane CH ₄	8%	12%	10%	13%	18%	7%
Ethane C ₂ H ₆	9%	11%	12%	17%	24%	7%
Ethene C ₂ H ₄	8%	12%	12%	12%	29%	7%
Ethyn C ₂ H ₂	9%	11%	12%	11%	35%	7%
Propane C ₃ H ₈	n.e.	11%	9%	n.e.	n.e.	n.e
Propene C ₃ H ₆	12%	n.e.	n.e.	26%	20%	n.e

Table 3: comparison of the relative uncertainties on the various components

The results of the 2018 PT show an improvement compared to the previous years, but did not meet the strict requirements of the target test method.

5. DISCUSSION

The consensus values as determined in this PT are compared with the average values from the homogeneity testing by Morgan Schaffer in the following table. From this comparison, it is clear that all consensus values as determined in this PT are very well in line with the values as determined by Morgan Schaffer after the preparation of the syringes.

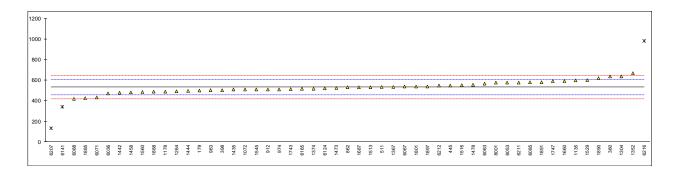
Component	Average values by Morgan Schaffer in µl/L	Consensus values from participants results in µl/L	Differences in µl/L	Calculated z-scores
Hydrogen H ₂	ogen H ₂ 518		15	0.39
Oxygen O ₂	15600	15499	-101	-0.09
Nitrogen N ₂ 53200		56117	2917	0.73
Carbon Monoxide CO 517		512	-5	-0.14
Carbon Dioxide CO ₂	arbon Dioxide CO ₂ 547		8	0.20
Methane CH ₄	Methane CH ₄ 518		0	0.00
Ethane C ₂ H ₆ 521		536	15	0.39
Ethene C ₂ H ₄ 514		524	10	0.27
Ethyn C ₂ H ₂ 519		510	-9	-0.25

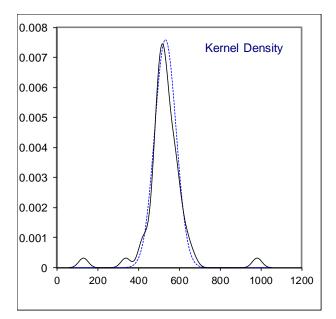
Table 4: comparison of consensus values with values determined by Morgan Schaffer

No statistical differences were observed between the test results of the 50mL syringes and the 100mL syringes. Both the averages and the variabilities for each of the components were in line with each other for both type of syringes. Regarding the extraction method used a vast majority of the participants (83%) used "Head Space".

Determination of Hydrogen (H₂) on sample #18234; results in µI/L

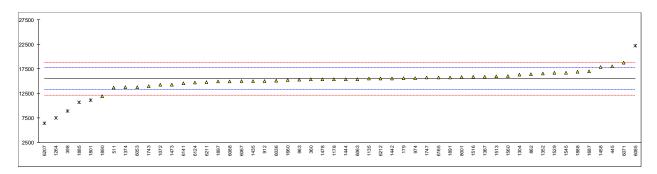
	mination of Hydrog				
lab 179	method D3612	value 499	mark	z(targ) -0.89	remarks
237	D3012	499		-0.89	
360	IEC60567	634.2		2.67	
398	IEC60567	503.1		-0.78	
445	IEC60567	546.8	С	0.37	first reported 393.4
511	D3612	533		0.01	
614					
862	IEC60567	531	_	-0.04	
912	D3612	510	С	-0.60	first reported 229
913 962					
963	D3612	502.7		-0.79	
974	D3612	510		-0.79	
1072	IEC60567	508.1		-0.65	
1135	IEC60567	597		1.69	
1178	IEC60567	488.6		-1.16	
1264	D3612	490		-1.12	
1304	INH-120	636.2		2.72	
1352	IEC60567	668.0		3.56	
1367 1374	D3612 D3612	533.51 516.4		0.02 -0.43	
1430	D3012	510.4		-0.43	
1435	IEC60567	508		-0.65	
1442	IEC60567	477.0		-1.46	
1444	IEC60567	496.50844		-0.95	
1458	D3612	481		-1.36	
1473	IEC60567	524.5		-0.22	
1478	IEC60567	552.99		0.53	
1513 1516	IEC60567	531.66 550.7		-0.03 0.47	
1529	IEC60567	600		1.77	
1545	D3612	510	С	-0.60	first reported 314.0
1560	IEC60567	485		-1.25	'
1660	IEC60567	590		1.51	
1687	IEC60567	531.1		-0.04	
1720	15000507			0.57	
1743 1747	IEC60567 IEC60567	511 588.53		-0.57 1.47	
1801	IEC60567	537.28		0.12	
1885	D3612	425	С	-2.83	first reported 191
1888	IEC60567	486.6		-1.21	
1890	IEC60567	616.90		2.21	
1891	IEC60567	579		1.22	
1897	IEC60567	538		0.14	
6015 6036	IEC60567	469		 -1.67	
6053	IEC60567	576		1.14	
6063	IEC60567	565.40		0.86	
6067	IEC60567	536.315		0.10	
6071		430		-2.70	
6085	D3612	578		1.19	
6088	IEC60567	416.85		-3.04	
6124	IEC60567	521	D(0.05)	-0.31	
6141	D3612	338	R(0.05)	-5.12	
6165 6207	IEC60567 IEC60567	516 130.39349	R(0.01)	-0.44 -10.57	
6211	GB/T17623	576.50	14(0.01)	1.15	
6212	D3612	546.647		0.37	
6216	IEC60567	983	R(0.01)	11.83	
8001	IEC60567	574.7	. ,	1.10	
	normality	OK			
	n	50			
	outliers	3			
	mean (n) st.dev. (n)	532.70 52.541			
	R(calc.)	147.11			
	st.dev.(IEC60567:11)	38.050			
	R(IEC60567:11)	106.54			

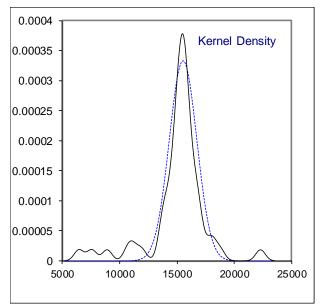




Determination of Oxygen (O2) on sample #18234; results in $\mu l/L$

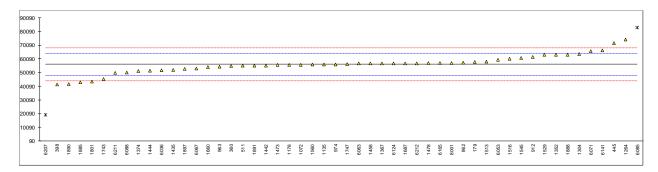
lab	method	value	mark	z(targ)	remarks
179	D3612	15585		0.08	
237	15000507	45044.0			
360 398	IEC60567 IEC60567	15344.2 8877	R(0.01)	-0.14 -5.98	
445	IEC60567	17991.2	C	2.25	first reported 31998
511	D3612	13680		-1.64	
614 862	IEC60567	16386.9		0.80	
912	D3612	15022	С	-0.43	first reported 5862
913					'
962	D0040	45007.0		0.45	
963 974	D3612 D3612	15327.9 15600		-0.15 0.09	
1072	IEC60567	14293.1		-1.09	
1135	IEC60567	15499		0.00	
1178 1264	IEC60567 D3612	15365.3 7545	R(0.01)	-0.12 -7.18	
1304	INH-120	16326.3	11(0.01)	0.75	
1352	IEC60567	16551.0		0.95	
1367 1374	D3612 D3612	15886.21 13740.2		0.35 -1.59	
1430	D3012	13740.2		-1.59	
1435	IEC60567	15008		-0.44	
1442	IEC60567	15552.0		0.05	
1444 1458	IEC60567 D3612	15403.5 17891		-0.09 2.16	
1473	IEC60567	14300.1		-1.08	
1478	IEC60567	15356.13		-0.13	
1513 1516	IEC60567	15993 15863.1		0.45 0.33	
1529	IEC60567	16700		1.09	
1545	D3612	16737.9		1.12	
1560	IEC60567	16051		0.50	
1660 1687	IEC60567 IEC60567	15225 16984.8		-0.25 1.34	
1720	1200007				
1743	IEC60567	14000		-1.35	
1747 1801	IEC60567 IEC60567	15729.57 11146.89	R(0.05)	0.21 -3.93	
1885	D3612	10700	C,R(0.05)	-4.33	first reported 11173
1888	IEC60567	16933.3	, ,	1.30	·
1890 1891	IEC60567 IEC60567	11897 15769		-3.25 0.24	
1897	IEC60567	14917		-0.53	
6015					
6036	IEC60567	15060		-0.40	
6053 6063	IEC60567 IEC60567	13763 15405.77		-1.57 -0.08	
6067	IEC60567	14991.49		-0.46	
6071		18731		2.92	
6085 6088	D3612 IEC60567	22249 14924	R(0.01)	6.10 -0.52	
6124	IEC60567	14718		-0.32	
6141	D3612	14546		-0.86	
6165 6207	IEC60567	15763 6410 27820	R(0.01)	0.24 -8.20	
6207 6211	IEC60567 GB/T17623	6419.27820 14768.64	N(U.U1)	-8.20 -0.66	
6212	D3612	15512.267		0.01	
6216	IFC60567	45040.2		0.22	
8001	IEC60567	15849.3		0.32	
	normality	suspect			
	n	46			
	outliers mean (n)	6 15498.74			
	st.dev. (n)	1199.291			
	R(calc.)	3358.02			
	st.dev.(IEC60567:11)	1107.053			
	R(IEC60567:11)	3099.75			

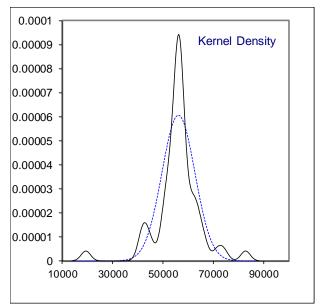




Determination of Nitrogen (N2) on sample #18234; results in $\mu l/L$

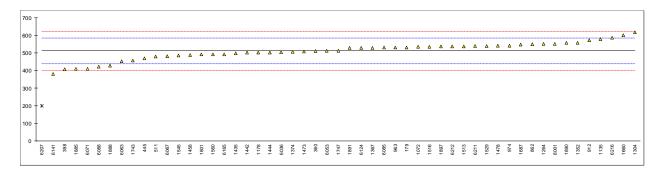
lab	method	value	mark	z(targ)	remarks
179	D3612	57952	man	0.46	
237	200.2				
360	IEC60567	54869.2		-0.31	
398	IEC60567	41457		-3.66	
445	IEC60567	71556.4	С	3.85	first reported 127287
511	D3612	55099		-0.25	•
614					
862	IEC60567	57262.3		0.29	
912	D3612	61474	С	1.34	first reported 23413
913					
962	_				
963	D3612	54355.3		-0.44	
974	D3612	56062		-0.01	
1072	IEC60567	55765.9		-0.09	
1135	IEC60567	56038 55607.7		-0.02	
1178 1264	IEC60567 D3612	55697.7 74158		-0.10 4.50	
1304	INH-120	63535.2		1.85	
1352	IEC60567	63015.0		1.72	
1367	D3612	56836.36		0.18	
1374	D3612	51305.1		-1.20	
1430					
1435	IEC60567	51915		-1.05	
1442	IEC60567	55236.0		-0.22	
1444	IEC60567	51545.9		-1.14	
1458	D3612	56805		0.17	
1473	IEC60567	55685.7		-0.11	
1478	IEC60567	56973.61		0.21	
1513	IEC60567	58025		0.48	
1516	.=	60316.8		1.05	
1529	IEC60567	63000		1.72	
1545	D3612	60728.7		1.15	
1560	IEC60567 IEC60567	55968 54232		-0.04 -0.47	
1660 1687	IEC60567	56888.9		0.47	
1720	IEC00307			0.19	
1743	IEC60567	45500		-2.65	
1747	IEC60567	56369.54		0.06	
1801	IEC60567	43554.18		-3.13	
1885	D3612	42895		-3.30	
1888	IEC60567	63128.1		1.75	
1890	IEC60567	41651		-3.61	
1891	IEC60567	55228		-0.22	
1897	IEC60567	52849		-0.82	
6015					
6036	IEC60567	51767		-1.09	
6053	IEC60567	59455		0.83	
6063	IEC60567	56649.55		0.13	
6067	IEC60567	53152.135		-0.74	
6071	D2612	65828	D(0.04)	2.42	
6085	D3612 IEC60567	82879	R(0.01)	6.68	
6088 6124	IEC60567	50065 56863		-1.51 0.19	
6141	D3612	66414		2.57	
6165	IEC60567	56991		0.22	
6207	IEC60567	19225.30000	R(0.01)	-9.20	
6211	GB/T17623	49818.34	(0.01)	-1.57	
6212	D3612	56904.3		0.20	
6216					
8001	IEC60567	57029.4		0.23	
	normality	suspect			
	n	50			
	outliers	2			
	mean (n)	56117.41			
	st.dev. (n)	6566.943			
	R(calc.)	18387.44			
	st.dev.(IEC60567:11)	4008.386			
	R(IEC60567:11)	11223.48			

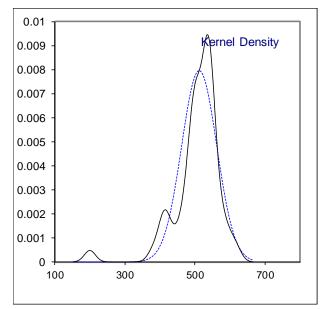




Determination of Carbon Monoxide (CO) on sample #18234; results in μ I/L

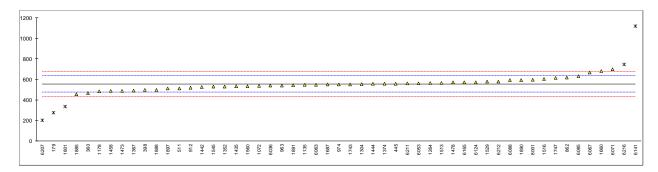
lab	method	value	mark	z(targ)	remarks
179	D3612	532	man	0.55	Tomarko
237	D0012				
360	IEC60567	511.1		-0.02	
398	IEC60567	408.6		-2.82	
445	IEC60567	469.5	С	-1.16	first reported 419.64
511	D3612	479		-0.90	
614					
862	IEC60567	549.9		1.04	
912	D3612	572	С	1.64	first reported 237
913					
962					
963	D3612	531.80		0.55	
974	D3612	541		0.80	
1072	IEC60567	536.1		0.66	
1135	IEC60567	578		1.81	
1178	IEC60567	502.96		-0.24	
1264	D3612	551		1.07	
1304	INH-120	618.0		2.90	
1352	IEC60567	559.0 530.05		1.29	
1367	D3612	530.05		0.50 -0.17	
1374	D3612	505.7		-0.17	
1430 1435	IEC60567	499		-0.35	
1435	IEC60567	502.5		-0.35 -0.26	
1444	IEC60567	503.18089		-0.24	
1458	D3612	489		-0.63	
1473	IEC60567	507.8		-0.11	
1478	IEC60567	540.87		0.79	
1513	IEC60567	538.54		0.73	
1516	1200007	536.4		0.67	
1529	IEC60567	540		0.77	
1545	D3612	486.7		-0.69	
1560	IEC60567	492		-0.54	
1660	IEC60567	601		2.44	
1687	IEC60567	547.0		0.96	
1720					
1743	IEC60567	457		-1.50	
1747	IEC60567	512.25		0.01	
1801	IEC60567	491.86		-0.55	
1885	D3612	409	С	-2.81	first reported 132
1888	IEC60567	429.3		-2.26	
1890	IEC60567	558.8		1.28	
1891	IEC60567	530		0.50	
1897	IEC60567	537		0.69	
6015 6036	IEC60567	505		-0.19	
6053	IEC60567	512		0.00	
6063	IEC60567	453.22		-1.60	
6067	IEC60567	481.185		-0.84	
6071	1200001	410		-2.79	
6085	D3612	531		0.52	
6088	IEC60567	421.37		-2.48	
6124	IEC60567	530		0.50	
6141	D3612	381		-3.58	
6165	IEC60567	492		-0.54	
6207	IEC60567	199.52482	R(0.01)	-8.54	
6211	GB/T17623	539.69	•	0.76	
6212	D3612	538.057		0.72	
6216	IEC60567	586		2.03	
8001	IEC60567	551.7		1.09	
					
	normality	OK			
	n ti	52			
	outliers	1			
	mean (n)	511.87			
	st.dev. (n)	50.181 140.51			
	R(calc.) st.dev.(IEC60567:11)	140.51 36.562			
	R(IEC60567:11)	102.37			
	14(12000001.11)	102.01			

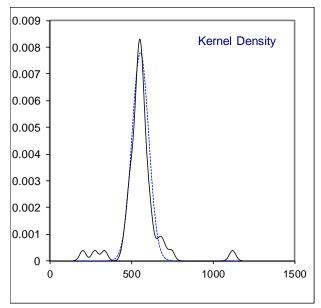




Determination of Carbon Dioxide (CO2) on sample #18234; results in $\mu l/L$

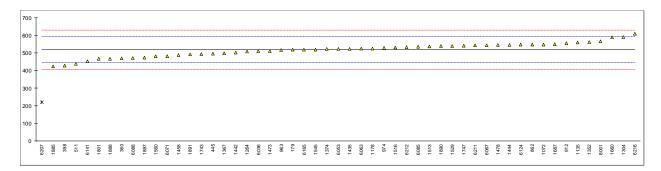
lab	method	value	mark	z(targ)	remarks
179	D3612	276	R(0.01)	-7.04	
237	_001L		11(0.01)	-7.04	
360	IEC60567	466.6		-2.23	
398	IEC60567	497.8		-1.44	
445	IEC60567	559.1	С	0.10	first reported 433.43
511	D3612	513		-1.06	'
614					
862	IEC60567	618.2		1.59	
912	D3612	519	С	-0.91	first reported 279
913					
962	_				
963	D3612	542.2		-0.32	
974	D3612	551		-0.10	
1072	IEC60567	536.2		-0.47	
1135 1178	IEC60567 IEC60567	548 485.6		-0.18 -1.75	
1264	D3612	564		0.23	
1304	INH-120	554.6		-0.01	
1352	IEC60567	531.0		-0.60	
1367	D3612	493.01		-1.56	
1374	D3612	557.5		0.06	
1430	200.2				
1435	IEC60567	533		-0.55	
1442	IEC60567	527.0		-0.71	
1444	IEC60567	556.92388		0.05	
1458	D3612	487		-1.71	
1473	IEC60567	487.6		-1.70	
1478	IEC60567	570.96		0.40	
1513	IEC60567	565		0.25	
1516	.= 0	603.9		1.23	
1529	IEC60567	580		0.63	
1545	D3612	528.3		-0.67	
1560	IEC60567 IEC60567	534 680		-0.53 3.15	
1660 1687	IEC60567	550.3		-0.12	
1720	IEC00307	550.5		-0.12	
1743	IEC60567	551		-0.10	
1747	IEC60567	613.34		1.47	
1801	IEC60567	334.57	R(0.05)	-5.56	
1885	D3612	455	C	-2.52	first reported 294
1888	IEC60567	497.9	-	-1.44	
1890	IEC60567	595.1		1.01	
1891	IEC60567	545		-0.25	
1897	IEC60567	512		-1.08	
6015					
6036	IEC60567	540		-0.38	
6053	IEC60567	563		0.20	
6063	IEC60567	548.58		-0.16	
6067	IEC60567	666.14		2.80	
6071	D2612	701		3.68	
6085	D3612	631		1.92	
6088 6124	IEC60567 IEC60567	592.77 574		0.95 0.48	
6141	D3612	574 1120	R(0.01)	14.25	
6165	IEC60567	573	13(0.01)	0.45	
6207	IEC60567	202.78964	R(0.01)	-8.88	
6211	GB/T17623	561.01	11(0.01)	0.15	
6212	D3612	580.968		0.66	
6216	IEC60567	745	R(0.05)	4.79	
8001	IEC60567	597.5	()	1.07	
	normality	suspect			
	n	48			
	outliers	5			
	mean (n)	554.98			
	st.dev. (n)	51.222			
	R(calc.)	143.42			
	st.dev.(IEC60567:11)	39.642			
	R(IEC60567:11)	111.00			

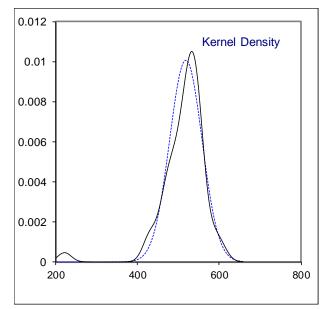




Determination of Methane (CH₄) on sample #18234; results in $\mu l/L$

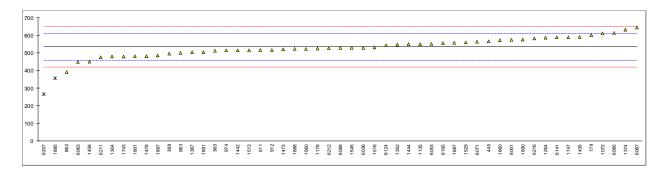
lab	method	value	mark	z(targ)	remarks
179	D3612	519	mark	0.02	
237	20012			0.02	
360	IEC60567	469.5		-1.32	
398	IEC60567	428.1		-2.44	
445	IEC60567	496.4	С	-0.59	first reported 467.69
511	D3612	439	O	-2.14	ilist reported 407.00
614	D3012				
862	IEC60567	547.2		0.78	
912	D3612	557	С	1.05	first reported 250
913	D3012		C	1.00	ilist reported 200
962					
963	D3612	516.00		-0.06	
974	D3612	529		0.29	
1072	IEC60567	548.3		0.23	
1135	IEC60567	561		1.15	
1178	IEC60567	526.17		0.21	
1264	D3612	509		-0.25	
1304	INH-120	591.1		1.97	
1352	IEC60567	563.0		1.21	
1367	D3612	498.49		-0.53	
1374	D3612	522.2		0.11	
1430	20012				
1435	IEC60567	524		0.16	
1442	IEC60567	502.0		-0.44	
1444	IEC60567	546.61044		0.77	
1458	D3612	488		-0.82	
1473	IEC60567	511.7		-0.18	
1478	IEC60567	544.81		0.72	
1513	IEC60567	536.77		0.50	
1516	1200007	531.2		0.35	
1529	IEC60567	540		0.59	
1545	D3612	519.1		0.02	
1560	IEC60567	482		-0.98	
1660	IEC60567	589		1.91	
1687	IEC60567	549.2		0.84	
1720					
1743	IEC60567	495		-0.63	
1747	IEC60567	541.65		0.63	
1801	IEC60567	467.36		-1.37	
1885	D3612	425	С	-2.52	first reported 207
1888	IEC60567	468.5	· ·	-1.34	
1890	IEC60567	539.3		0.57	
1891	IEC60567	492		-0.71	
1897	IEC60567	473		-1.22	
6015					
6036	IEC60567	510		-0.22	
6053	IEC60567	523		0.13	
6063	IEC60567	525.28		0.19	
6067	IEC60567	543.47		0.68	
6071		483		-0.95	
6085	D3612	536		0.48	
6088	IEC60567	470.89		-1.28	
6124	IEC60567	547		0.78	
6141	D3612	454		-1.74	
6165	IEC60567	519		0.02	
6207	IEC60567	220.77907	R(0.01)	-8.04	
6211	GB/T17623	543.39	(/	0.68	
6212	D3612	532.481		0.38	
6216	IEC60567	609		2.45	
8001	IEC60567	566.2		1.30	
		'			
	normality	OK			
	n	52			
	outliers	1			
	mean (n)	518.26			
	st.dev. (n)	39.674			
	R(calc.)	111.09			
	st.dev.(IEC60567:11)	37.018			
	R(IEC60567:11)	103.65			
	,				

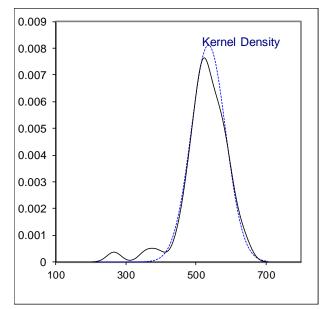




Determination of Ethane (C_2H_6) on sample #18234; results in $\mu I/L$

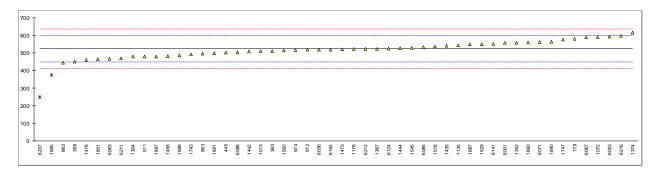
lab	method	value	mark	z(targ)	remarks
179	D3612	602		1.73	
237					
360	IEC60567	512.0		-0.62	
398	IEC60567	496.4		-1.03	
445	IEC60567	566.6	С	0.80	first reported 528.9
511	D3612	516		-0.52	·
614					
862	IEC60567	391.8		-3.76	
912	D3612	517	С	-0.49	first reported 245
913					
962					
963	D3612	500.30		-0.93	
974	D3612	514		-0.57	
1072	IEC60567	611.3		1.97	
1135 1178	IEC60567 IEC60567	550 524.93		0.37 -0.29	
1264	D3612	524.93 587		1.34	
1304	INH-120	480.4		-1.45	
1352	IEC60567	548.0		0.32	
1367	D3612	504.24		-0.83	
1374	D3612	633.2		2.54	
1430					
1435	IEC60567	592		1.47	
1442	IEC60567	515.5		-0.53	
1444	IEC60567	549.34569		0.35	
1458	D3612	452		-2.19	
1473	IEC60567	520.1		-0.41	
1478	IEC60567	482.67		-1.39	
1513	IEC60567	515.91		-0.52	
1516	15000507	531.0		-0.13	
1529	IEC60567	560 538.7		0.63	
1545 1560	D3612 IEC60567	528.7 524		-0.19 -0.31	
1660	IEC60567	573		0.97	
1687	IEC60567	557.5		0.56	
1720	1200007				
1743	IEC60567	481		-1.43	
1747	IEC60567	588.94		1.39	
1801	IEC60567	481.73		-1.41	
1885	D3612	357	C,R(0.05)	-4.67	first reported 207
1888	IEC60567	523.5		-0.32	
1890	IEC60567	575.7		1.04	
1891	IEC60567	505		-0.81	
1897	IEC60567	486		-1.30	
6015	.=				
6036	IEC60567	529		-0.18	
6053	IEC60567	553		0.45	
6063	IEC60567	449.82		-2.25	
6067 6071	IEC60567	643.955		2.82 0.76	
6085	D3612	565 613		2.01	
6088	IEC60567	528.52		-0.19	
6124	IEC60567	544		0.13	
6141	D3612	588		1.36	
6165	IEC60567	556		0.53	
6207	IEC60567	265.89513	R(0.01)	-7.05	
6211	GB/T17623	476.54		-1.55	
6212	D3612	527.478		-0.22	
6216	IEC60567	582		1.20	
8001	IEC60567	575.2		1.03	
	normality	OK			
	n	51			
	outliers	2			
	mean (n)	535.89			
	st.dev. (n)	49.110			
	R(calc.)	137.51			
	st.dev.(IEC60567:11)	38.278 107.18			
	R(IEC60567:11)	107.18			

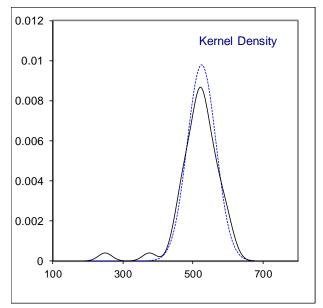




Determination of Ethene / Ethylene (C_2H_4) on sample #18234; results in $\mu I/L$

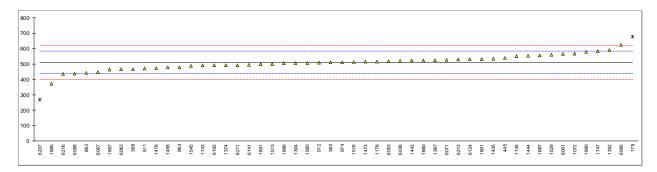
lab	method	value	mark	z(targ)	remarks
179	D3612	580	········	1.49	
237	_001L				
360	IEC60567	511.5		-0.34	
398	IEC60567	450.4		-1.97	
445	IEC60567	503.3	С	-0.56	first reported 497.3
511	D3612	480		-1.18	•
614					
862	IEC60567	444.7		-2.13	
912	D3612	520	С	-0.12	first reported 303
913					
962	_				
963	D3612	496.10		-0.75	
974	D3612	517		-0.20	
1072	IEC60567	591.7		1.80	
1135 1178	IEC60567 IEC60567	543 522.56		0.50 -0.05	
1264	D3612	522.56	W	-0.05	first reported 501
1304	INH-120	479.3	VV	-1.20	ilist reported 50 i
1352	IEC60567	559.0		0.92	
1367	D3612	523.99		-0.01	
1374	D3612	615.9		2.44	
1430	- 				
1435	IEC60567	541		0.44	
1442	IEC60567	508.5		-0.42	
1444	IEC60567	528.21157		0.10	
1458	D3612	483		-1.10	
1473	IEC60567	521.9		-0.07	
1478	IEC60567	462.09		-1.66	
1513	IEC60567	509.86		-0.39	
1516	15000507	537.8		0.36	
1529	IEC60567	550 530.8		0.68	
1545 1560	D3612	529.8		0.15	
1560 1660	IEC60567 IEC60567	515 561		-0.25 0.98	
1687	IEC60567	549.4		0.96	
1720	12000001	549.4		0.07	
1743	IEC60567	493		-0.84	
1747	IEC60567	576.90		1.40	
1801	IEC60567	466.08		-1.56	
1885	D3612	376	C,R(0.05)	-3.96	first reported 294
1888	IEC60567	486.2	,	-1.02	·
1890	IEC60567	563.8		1.05	
1891	IEC60567	498		-0.70	
1897	IEC60567	481		-1.16	
6015					
6036	IEC60567	520		-0.12	
6053	IEC60567	594		1.86	
6063	IEC60567	467.57		-1.52	
6067	IEC60567	588.39		1.71	
6071	D2612	562		1.00	
6085 6088	D3612 IEC60567	533 505.27		0.23 -0.51	
6124	IEC60567	505.27 526		0.04	
6141	D3612	552 552		0.04	
6165	IEC60567	520		-0.12	
6207	IEC60567	250.35796	R(0.01)	-7.32	
6211	GB/T17623	470.17	(0.01)	-1.45	
6212	D3612	523.346		-0.03	
6216	IEC60567	598		1.97	
8001	IEC60567	557.7		0.89	
	normality	OK			
	n	50			
	outliers	2			
	mean (n)	524.37			
	st.dev. (n)	40.699			
	R(calc.)	113.96			
	st.dev.(IEC60567:11)	37.455			
	R(IEC60567:11)	104.87			

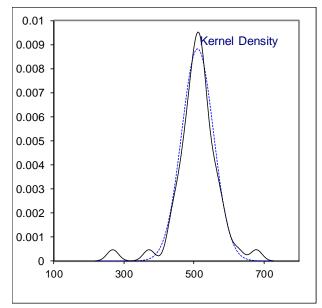




Determination of Ethyn /Acetylene (C_2H_2) on sample #18234; results in $\mu I/L$

lab	method	value	mark	z(targ)	remarks
179	D3612	677	R(0.05)	4.58	-
237	IE000507	 544 O		0.00	
360 398	IEC60567 IEC60567	511.0 466.4		0.02 -1.20	
445	IEC60567	539.1	С	0.79	first reported 502.8
511	D3612	470		-1.10	
614 862	IEC60567	 442.5		 -1.86	
912	D3612	508	С	-0.06	first reported 186
913					
962	D0040	470.00			
963 974	D3612 D3612	478.30 512		-0.87 0.05	
1072	IEC60567	566.9		1.56	
1135	IEC60567	552		1.15	
1178 1264	IEC60567 D3612	516.74 	W	0.18	first reported 482
1304	INH-120	505.7	VV	-0.12	mst reported 402
1352	IEC60567	592.0		2.25	
1367	D3612	525.26	0	0.41	Continuo anta di OAA 7
1374 1430	D3612	493 	С	-0.47 	first reported 641.7
1435	IEC60567	535		0.68	
1442	IEC60567	522.0		0.32	
1444	IEC60567	553.94086		1.20	
1458 1473	D3612 IEC60567	477 515.3		-0.91 0.14	
1478	IEC60567	474.36		-0.98	
1513	IEC60567	498.50		-0.32	
1516 1529	IEC60567	513.4 560		0.09 1.37	
1545	D3612	486.4		-0.65	
1560	IEC60567	507		-0.09	
1660	IEC60567	523		0.35	
1687 1720	IEC60567	555.9 		1.26	
1743	IEC60567	492		-0.50	
1747	IEC60567	583.32		2.01	
1801 1885	IEC60567 D3612	533.075 372	С	0.63 -3.79	first reported 288
1888	IEC60567	505.1	C	-0.14	mst reported 200
1890	IEC60567	579.3		1.90	
1891	IEC60567	498		-0.33	
1897 6015	IEC60567	463 		-1.29 	
6036	IEC60567	520		0.27	
6053	IEC60567	519		0.24	
6063 6067	IEC60567 IEC60567	465.66 447.76		-1.22 -1.71	
6071	12000307	528		0.49	
6085	D3612	624		3.12	
6088	IEC60567	437.97		-1.98	
6124 6141	IEC60567 D3612	532 494		0.60 -0.44	
6165	IEC60567	492		-0.50	
6207	IEC60567	268.91675	R(0.01)	-6.62	
6211 6212	GB/T17623 D3612	493.18 528.919		-0.47 0.51	
6216	IEC60567	434.9		-2.07	
8001	IEC60567	564.1		1.48	
	normality	suspect			
	n outliers	50			
	mean (n)	2 510.16			
	st.dev. (n)	45.188			
	R(calc.)	126.53			
	st.dev.(IEC60567:11) R(IEC60567:11)	36.440 102.03			
	(1200007.71)	.02.00			



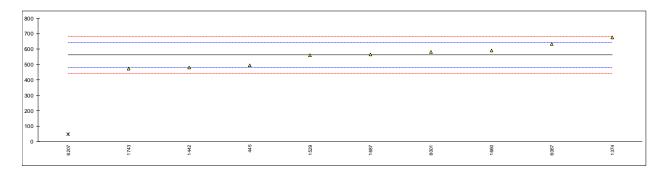


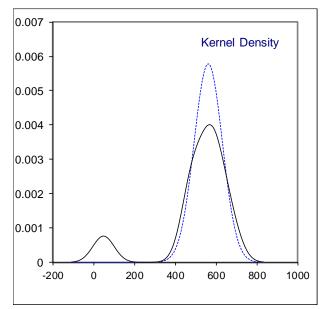
Determination of Propane (C_3H_8) on sample #18234; results in $\mu I/L$

lab	method	value	mark	z(targ)	remarks
179					
237					
360					
398 445					
511					
614					
862					
912	D3612	540	ex,C		first reported 297, possibly false positive test result? ex see § 4.1
913					
962 963					
974	D3612	458	ex,C		first reported 518, possibly false positive test result? ex see § 4.1
1072			•		
1135					
1178 1264					
1304					
1352	IEC60567	625.0	ex		possibly false positive test result? ex see § 4.1
1367					
1374	D3612	0.5			
1430 1435					
1435	IEC60567	<1			
1444	1200007				
1458					
1473					
1478					
1513 1516					
1529	IEC60567	0.82			
1545					
1560					
1660	IEC60567	1			
1687 1720	IEC60567	2.0			
1743	IEC60567	0.16			
1747	IEC60567	Nil			
1801					
1885					
1888 1890					
1891					
1897					
6015					
6036					
6053 6063					
6067	IEC60567	0	С		first reported 670.745
6071					
6085	D3612	705	ex		possibly false positive test result? ex see § 4.1
6088 6124					
6124	D3612	609	ex		possibly false positive test result? ex see § 4.1
6165			- A		
6207	IEC60567	0.0			
6211	D0040		147		Cont. con ant of 505.00
6212	D3612		W		first reported 535.68
6216 8001	IEC60567	0			
3001					
	normality	not OK			
	n outliers	10 0 +5ex			
	mean (n)	<10			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	st.dev.(IEC60567:11)	n.a.			
	R(IEC60567:11)	n.a.			

Determination of Propene (C_3H_6) on sample #18234; results in $\mu I/L$

lab	method	value	mark	z(targ)	remarks
179					
237					
360					
398					
445	IEC60567	493.9		-1.69	
511					
614 862					
912					
913					
962					
963					
974					
1072					
1135					
1178					
1264					
1304					
1352					
1367 1374	D3612	675.5		2.84	
1430	D0012	073.3 		2.04	
1435					
1442	IEC60567	479.5	С	-2.04	first reported 279.5
1444					•
1458					
1473					
1478					
1513					
1516 1529	IEC60567	560		-0.04	
1545	ILC00307			-0.04	
1560					
1660	IEC60567	591		0.74	
1687	IEC60567	565.8		0.11	
1720					
1743	IEC60567	474		-2.18	
1747	IEC60567		W		first reported Nil
1801 1885					
1888					
1890					
1891					
1897					
6015					
6036					
6053					
6063 6067	IEC60567	632.514	С	1.77	first reported 0
6071	1200001	032.514	C	1.77	iii at reporteu o
6085					
6088					
6124					
6141					
6165	15000507	40.05005	D(0.04)	40.70	
6207	IEC60567	48.65925	D(0.01)	-12.79 	
6211 6212					
6216					
8001	IEC60567	581.4		0.50	
	normality	OK			
	n	9			
	outliers	1			
	mean (n)	561.51 60.170			
	st.dev. (n) R(calc.)	69.170 193.68			
	st.dev.(IEC60567:11)	40.108			
	R(IEC60567:11)	112.30			
	` '				





Analytical details

	T (Fortunation
lab	Type of syringe (mL)	Extraction method
179	100	Stripper Column
237 360	100	Head Space
398	50	Head Space
445	50	Head Space
511	50	Head Space
614		·
862		
912		
913		
962 963	 50	Head Space
974	100	Head Space
1072	100	Toepler
1135	50	Head Space
1178	100	Head Space
1264	100	Head Space
1304	50	Head Space
1352	100	TOGA/GC system
1367	100	Head Space
1374 1430	50 	Head Space
1435	50	Head Space
1442	50	Head Space
1444		
1458	100	Stripper Column
1473	50	Head Space
1478	100	Toepler
1513	100	Toepler
1516	50 100	Head Space
1529 1545	100 100	Head Space Head Space
1560	100	Head Space
1660	50	Head Space
1687	100	Head Space
1720		
1743	100	Head Space
1747	50	Head Space
1801	50 100	Head Space
1885 1888	100 	Toepler
1890	100	Head Space
1891	50	Head Space
1897	50	Head Space
6015		
6036	50	Head Space
6053		
6063	100	Toepler
6067 6071	100 	Head Space
6085	50	Head Space
6088	100	Head Space
6124	50	Stripper Column
6141	100	Head Space
6165	50	Head Space
6207	100	Head Space
6211	100	Head Space
6212 6216	100	Head Space
8001	50 100	Head Space Head Space
3001	100	

Number of participants per country

- 7 labs in AUSTRALIA
- 3 labs in BELGIUM
- 1 lab in BULGARIA
- 2 labs in CHINA, People's Republic
- 1 lab in CROATIA
- 1 lab in FRANCE
- 3 labs in GERMANY
- 1 lab in GREECE
- 1 lab in HONG KONG
- 3 labs in INDIA
- 2 labs in ITALY
- 1 lab in KINGDOM OF BAHRAIN
- 1 lab in MALAYSIA
- 1 lab in MEXICO
- 1 lab in MOROCCO
- 1 lab in NETHERLANDS
- 1 lab in NIGERIA
- 1 lab in PERU
- 2 labs in PORTUGAL
- 1 lab in QATAR
- 4 labs in SAUDI ARABIA
- 3 labs in SINGAPORE
- 2 labs in SLOVENIA
- 1 lab in SOUTH KOREA
- 5 labs in SPAIN
- 1 lab in SUDAN
- 1 lab in SWITZERLAND
- 3 labs in UNITED ARAB EMIRATES
- 3 labs in UNITED KINGDOM
- 1 lab in UNITED STATES OF AMERICA
- 1 lab in URUGUAY

Abbreviations:

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

D(0.01) = outlier in Dixon's outlier test D(0.05) = straggler in Dixon's outlier test D(0.01) = outlier in Grubbs' outlier test D(0.05) = straggler in Grubbs' outlier test D(0.05) = outlier in Double Grubbs' outlier test D(0.05) = straggler in Double Grubbs' outlier test

R(0.01) = outlier in Rosner's outlier test R(0.05) = straggler in Rosner's outlier test E = possibly an error in calculations

ex = test result excluded from statistical evaluations

n.a. = not applicable n.e. = not evaluated fr. = first reported

SDS = Safety Data Sheet

Literature:

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- 11 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
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- 14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
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