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Liquid petroleum products — Middle distillates and fatty acid methyl ester (FAME) fuels and blends — Round Robin report on applicability of Rapid Small Scale Oxidation Test method

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Liquid petroleum products - Middle distillates and fatty acid methyl ester (FAME) fuels and blends - Round Robin report on applicability of Rapid Small Scale Oxidation Test method

Produits pétroliers liquides - Carburants et mélanges des distillats moyens et des esters méthyliques d'acides gras (EMAG) - Réport de Round Robin de l'application de la détermination methode d'oxydation accéléree petite échelle

Flüssige Petroleum Produkte - Mitteldestilat und Fettsäuremethylester (FAME) Kraftstoffen und Mischungen - Round Robin Rapport der Applikation des beschleunigten kleinen Maßstaben Oxidationstests

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Foreword

This document (CEN/TR 16366:2012) has been prepared by Technical Committee CEN/TC 19 “Gaseous and Liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

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This document presents background to EN 16091.

1 Scope

This Technical Report describes a series of round robin test campaigns for precision estimation of EN 16091. Furthermore, this document includes a summary of the results of the RRTs (see Clause 7 and Annex B).

NOTE The identities of the participating laboratories are not displayed in this report. They are, however filed with the original RRT documentation at the CEN/TC 19/JWG1 secretariat.

2 Test method background

The Rapid Small Scale Oxidation Test method (RSSOT[1], EN 16091, [1]) is developed as an additional or parallel test method for the determination of oxidation stability (EN 14112 [2] and EN 15751 [3]) in FAMEs according to EN 14214 [5] and diesel fuel – biodiesel blends [4].

Three precision studies (2007-231, 2008-231, 2010-231) have been executed in CEN/TC 19/JWG 1 in order to access the test method precision in terms of repeatability, *r,* and reproducibility, *R*.

NOTE In addition, more RR-testing has been executed in order to compare results with those from other tests like EN 14112, EN 15751 or acid number after ageing. These additional test results are not subject of this precision report but are reported elsewhere. It shall be noted that the Rancimat[2] and PetroOXY[1] results exhibit a good correlation, obviously indicating that both methods provide good estimations of oxidation stability.

The three precision studies (2007-231, 2008-231, 2010-231) contained the following:

— RRT 2007-231 → pre- study with 10 samples (“B2” .. “B30”) in 5 laboratories;

— RRT 2008-231 → RRT with 23 samples (“B0” .. “B100”) in 19 laboratories;

— RRT 2010-231 → RRT with 7 samples testing the influence of EHN in 11 labs (EHN = Cetane improver = 2-ethyl hexyl nitrate).

The draft version of EN 16091 was sent to the laboratories as the requested test procedure along with the Round Robin instructions. The same test method procedure was used is the three different RRTs. Since no significant modifications had been made which could be precision-relevant, this test procedure remained the same as specified in the final standard (EN 16091:2011).

3 Description of the samples

3.1 RRT 2007-231

There were 10 samples used for the pre-study in 2007. Each sample was randomized, blind coded and distributed by the coordinator of this RRT.

The following samples had been agreed by the CEN/TC 307/WG 1, 10 Bx samples: 1 x B2, 1 x B4, 1 x B5, 1 x B7, 1 x B8, 1 x B12, 1 x B15, 1 x B20, 1 x B30, 1 x B100. The samples have been prepared by blending of one B0 and one B100 (RME/SME (90/10)). Information of the fuel properties are not provided.

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1) Also known as the PetroOXY test. PetroOXY is the trade name of a product supplied by Petrotest, Instruments GmbH & Co, Germany. This information is given for the convenience of users of this European Technical Report and does not constitute an endorsement by CEN of the product named.

2) Rancimat is the trade name of a product, model 743, supplied by Metrohm AG, Switzerland and an example of suitable equipment available comercially. This information is given for the convenience of users of this European Technical Report and does not constitute an endorsement by CEN of the product named.

3.2 RRT 2008-231

There were 23 samples used for the RRT 2008-231. Each sample was randomized, blind coded and distributed by the coordinator of this RRT.

The following samples were agreed by the CEN/TC 307/WG 1, in addition to 4 B100 Samples 19 Bxx samples were used: 3 x B00, 6 x B05, 6 x B10 and 4 x B30. The 16 Bxx (B05, B10, B30) fuels have been prepared by blending of one B00 and one B100 without the addition of other products (additives).

The properties of B0 are given in Table 1.

Table 1 — Diesel Fuel B0 properties for the RRT 2008-231

Property	Unit	Method	Fuel 1[a] EN 590 fuel[d]	Fuel 2[b] EN 590 fuel[d]	Fuel 3[c] Swedish Class 1[d]
Sulfur Content	mg/kg	EN 20846	8,6	9,8	< 3
FAME Content	% (*V/V*)	EN 14078	0	0,2	0
PAC	% (*m/m*)	EN 12916	5,5	2,4	0,1

[a] Additised with lubricity- and CFPP improver — no cetane improver
[b] EHN (Ethyl – Hexyl – Nitrate) < 100 mg/kg (IR test method)
[c] No information
[d] The suppliers of these fuels are available from the Secretariat of CEN/TC 19/JWG 1.

The properties of FAME (B100) are given in Table 2.

Table 2 — FAME (B100) properties for RRT 2008-231

Parameter	Unit	Method	FAME A[a]	FAME B[a]	FAME C[a]	FAME D[a]
Composition			RME	RME	SME 75 % PME 25 %	RME 60 %
BHT	mg/kg	—	No	1 000	No	No
Oxidation stability	h	EN 15751	5,8	13,6	7,2	6,0

[a] The supplier of all FAMEs is available from the Secretariat of CEN/TC 19/JWG 1.

The sample identification is given in Table 3.

Table 3 — Samples identification RRT 2008-231

Sample Number	Fuel Type	Sample Code	Diesel fuel used	FAME used	FAME content (% (*V/V*))
S1	B100	FAME A	—	—	100
S2	B100	FAME B	—	—	100
S3	B100	FAME C	—	—	100
S4	B100	FAME D	—	—	100
S5	B10	G1A10	1	A	10
S6	B30	G1A30	1	A	30
S7	B5	G1A5	1	A	5
S8	B10	G1B10	1	B	10
S9	B30	G1B30	1	B	30
S10	B5	G1B5	1	B	5
S11	B10	G1C10	1	C	10
S12	B30	G1C30	1	C	30
S13	B5	G1C5	1	C	5
S14	B10	G1D10	1	D	10
S15	B30	G1D30	1	D	30
S16	B5	G1D5	1	D	5
S17	B10	G2B10	2	B	10
S18	B10	G2A10	2	A	5
S19	B5	G2C5	2	C	5
S20	B5	G2D5	2	D	5
S21	B0	Fuel No.1	—	—	—
S22	B0	Fuel No.2	—	—	—
S23	B0	Fuel No.3	—	—	—

3.3 RRT 2010-231

There were in origin 7 samples in the RRT 2010-231: 5 x B0 samples without Ignition Improver.

The suppliers of these fuels are available from the secretariat of CEN/TC 19/JWG 1. All B0 samples meet the requirements of EN 590. The properties are given in Table 4.

Table 4 — Diesel fuels B0 properties

Property	Unit	Method	B01[a] Swedish Class 1	B02[a] EN 590	B03[a] EN 590	B04[a] EN 590	B05[a] EN 590
Sulphur	mg/kg	EN 20884	< 1	N/A	4,7	< 3	12
FAME	% (*V*/*V*)	EN 14078	< 0,05	N/A	< 0,1	< 0,1	< 0,5
PAH	% (*m*/*m*)	EN 12916	0,1	N/A	4,8	1,4	2,1
IBP	°C	ISO 3405	N/A	N/A	189	207,9	200,1
T95	°C	ISO 3405	284	N/A	363	327,3	352,2
Flashpoint	°C	EN 2719	72	N/A	80,5	85	81,5
[a] The suppliers of these fuels are available from the secretariat of CEN/TC 19/JWG 1.							

In addition two B7 samples were used. One from a local station and one based on the B04 EN 590 sample blended with RME up to a content of 7 % RME.

The B0 and B7 samples were prepared with different concentrations of EHN (EHN = Cetane improver = 2-ethyl hexyl nitrate), and the following sample set was used in eleven labs (see Table 5).

Table 5 — Sample set used

FUEL	EHN 0 mg/kg	EHN 500 mg/kg	EHN 1000 mg/kg
B01	X	X	X
B02	X	X	X
B03	X	X	X
B04	X	X	X
B05	X	X	X
B7-1	X	X	—
B7-2	X	X	—

In total, there were 19 samples. Each sample was randomized, blind coded and distributed by the coordinator of this RRT.

For each RRT and laboratory participant a verification fluid from the manufacturer was sent to check if the used instruments were in the limit of a given oxidation stability value.

4 Round Robin Instructions

The test program instructions were sent to each participating laboratory (see Annex A).

5 Description of equipment

The apparatus used by each laboratory in this study to develop a precision statement was the Petrotest, model PetroOXY[1)].

6 Form of data reports

Each laboratory was provided with a data report form for collection of data. The filled out data report forms were sent by the laboratories to the coordinator of these RRTs.

7 Statistical data summary

All precision values have been calculated following the procedures in EN ISO 4259 [6]. For the final precision statement in EN 16091, the results from RRT 2008-231 and 2010-231 were combined, resulting in the following precision formulae:

$$r = 0{,}288\,X + 0{,}496\ 5 \tag{1}$$

$$R = 0{,}086\ 3X + 1{,}377\ 2 \tag{2}$$

where

r is the repeatability (the difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material);

R is the reproducibility (the difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material);

X represents the mean of the two results expressed in min, rounded to the nearest 0,01 min.

Some more statistical detail and data is given in Annex B. The statistical review was provided by several precision experts of DIN FAM (Fachausschuss Mineralöl- und Brennstoffnormung des NMP (FAM)).

Annex A
(informative)

Comparative study protocol for oxidation stability test methods

IMPORTANT — Before starting sample measurements, read this protocol carefully and follow the instructions.

A.1 Scope and overview

The main purpose of this CEN Round Robin is to determine the precision of three proposed 'Oxidation Stability' test methods: EN 15751 (Rancimat[2)]) method, JAMA (Delta Acid Number) method and PetroOXY[1)] method. A second objective is to determine if any correlations exist between any of the measured properties of the methods.

The specific consideration of this study is to find a useful method which is able to predict the oxidation stability for pure mineral oil diesel (B0) and different bio-diesel blends (B5, B10, B30). The Rancimat and PetroOXY methods will also make measurements on pure FAME (B100) materials.

Precision will be determined in accordance with EN ISO 4259. The correlations between methods will be determined by additional statistical means.

A.2 Round Robin sample reception

Along with the PetroOXY test method protocol (dated XX/XX/XX) you will receive:

— a set of 23 samples with unique codes;

— 50 plastic pipettes;

— 100 O–ring seals;

— 1 Petrotest verification fluid;

— an instruction manual.

You will be provided with a unique laboratory code to preserve confidentiality. The laboratory code and the designated sample number and its origin are known only to the round robin programme coordinator.

A computer programme has randomly generated the testing order and no two laboratories have the same order. Upon receipt of the samples, please do the following:

— unpack the samples from the box;

— check the samples containers for any signs of damage or leakage;

— if there is any, please contact the programme manager and add all information indicated on the label glued to the sample container;

— as soon as possible, store the samples away from direct sunlight and less than +25 °C is recommended.

A.3 Test conditions for the round robin

Please refer to the attached copy of the short instruction manual (13-3000K.doc [3)]).

Settings for the round robin tests:

— test program: DIESEL;

— test temperature: 140 °C;

— filling pressure: 700 kPa (Oxygen);

— sample amount: 5 ml;

— remove the previous sample by means of a pipette or similar device;

— remove the used “O-ring” seal after each test and discard.

NOTE To avoid contamination of the new test, it is necessary to discard the used “Oring” seal, because it might be soaked with oxidation products from the previous test.

IMPORTANT — Only the O-ring seals delivered together with the test samples shall be used for the test.

As a solvent for the removal of oxidation residues from the test vessel ethanol of approx. 95 % (*V/V*) commercial available has been found to be suitable and shall be used.

The solvent shall be of suitable purity to leave no residue on the apparatus. Wipe the test sample cup, the seal groove and the cover of the test vessel with lint-free cleaning tissue soaked with solvent until free of gum or other oxidation residues. Allow the test sample cup and cover to dry in air and visually inspect for cleanliness. Compressed air is generally unsuitable to speed up evaporation of solvent because it can contain traces of oil that could contaminate the next test.

IMPORTANT — Insert a new “O-ring” seal for every new test.

Using a new pipette which you have received together with the round robin samples for every new test, place 5 ml ± 0,5 ml of the sample into the test sample cup.

Proceed further according the short instruction manual[3)].

A.4 Testing

Please ensure that test methods and manufacturers instructions are carefully followed.

Make sure that the sample temperature is approximately ambient temperature prior opening the sample container.

Before starting the testing of RR samples, run a test with the Petrotest verification fluid which is enclosed with the sample set.

IMPORTANT — This verification fluid shall be tested prior starting the tests with the RR samples.

Make sure that the obtained result is within 1 h and 57 min ± 6 min.

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3) The short instruction manual is available from the secretariat of CEN/TC 19/JWG 1 or Petrotest (sales@petrotest.com).

If the result is outside the limits, then repeat the test with a fresh portion of the verification fluid.

Report the value of the verification fluid in the RR protocol.

If the result is still out of limits – contact Petrotest at […...]

NOTE Only one bottle of Petrotest verification fluid is supplied. The main point is to ensure the equipment is in quality control.

A.5 Running the samples

Only two tests shall be carried out on each sample and both results shall be included on the test results sheet.

— using fresh sample each time;

— same Operator;

— same equipment;

— new O-ring;

— new pipette for sample filling.

On each sample container you will find the Sample ID Code.

Run the samples in the testing order as stated by the Programme Manager.

It is quite important to run all tests on the same instrument with the same operator and as soon as possible after the first test (repeatable conditions).

In any case report the results of two consecutive runs on each sample.

The induction period shall be reported for each test in:

hours:minutes:seconds

TIME FRAME: 4 weeks maximum after receipt of the sample set.

After completion of the Round Robin send the report form to:

{……..}

Thank you for your participation in this Round Robin.

Annex B
(informative)

Precision estimation for PetroOxy test

B.1 General

This annex includes the evaluations on RRT 2007-231, RRT 2008-231 and RRT 2010-231. Responsible for the RRTs was CEN/TC 19/JWG 1.

NOTE CEN/TC 19/JWG 1 was established in 2010. Responsible for the RRT in 2007 and 2008 was CEN/TC 19-CEN/TC 307 JWG, the predecessor of CEN/TC 19/JWG 1.

B.2 Methods and calculation used

All calculations were executed separately for each of the three exercises following the procedures, calculations and outlier tests from EN ISO 4259 [6].

Transformations were not executed because an evenly spaced set of test results over the measurement range could not be accomplished.

For the final precision estimation, the results from RRT 2008-231 and RRT 2010-231 were combined into one precision statement. Since the block matrix resulting from the combination of the two separate RRTs has only very little overlap, the precision functions $r = f(x)$ and $R = f(x)$ were calculated from the combined triplets (mean, *r*, *R*) as described below.

Since no simple recipe was found to do outlier tests on the block matrix described above, alternative outlier criteria had to be agreed, which were:

— accept (exclude) outliers as indicated in the two segregated RRT exercises;

— exclude samples with test results outside the agreed measurement range;

— exclude samples for which the reproducibility, R, is much higher than the reproducibility from the (sorted) neighbour samples.

Separate and combined results are described and discussed on the following pages.

B.3 RRT 2007-231 – pre-study

This clause gives an overview over the results of the pre-study RRT 2007-231. For this pre-study 10 samples in the range from “B2” to “B100” were used. Table B1 gives an overview over the oxidation stability of the samples tested and the corresponding values for *r, R* and *R*%. (“*R*%” is the relative Reproducibility as percentage from the test result, calculated as 100 * *R / X*).

Table B.1 — Results of the pre study RRT 2007-231

Sample	Oxidation stability min	*r*	*R*	*R* %
1	25,90	1,299	8,184	31,6
2	37,57	3,582	3,386	9,0
3	38,03	1,246	7,108	18,7
4	64,44	1,642	18,193	28,2
5	52,09	2,109	1,556	3,0
6	48,14	1,366	1,669	3,5
7	68,43	2,623	11,152	16,3
8	48,50	1,578	9,291	19,2
9	14,19	0,347	3,796	26,8
10	80,44	1,339	15,982	19,9

Figure B.1 displays a good relationship between the accelerated oxidation method according to the modified Rancimat, EN 15751 (X-axis, hours), and the PetroOXY test according to EN 16091 (Y-Axis, minutes), clearly depicted by the corresponding 95 % confidence- and prediction bands.

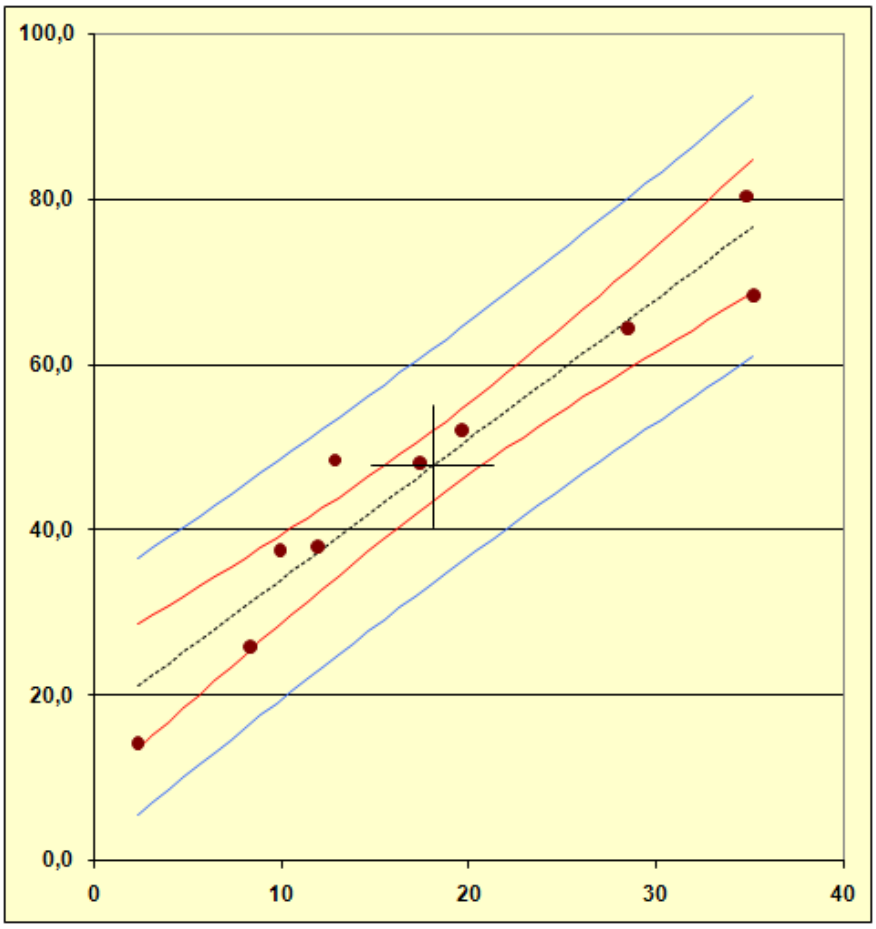


Figure B.1 — Comparison of the results of EN 15751 and EN 16091

The following conclusions could be drawn from the above mentioned results:

a) the results of the PetroOXY test according to EN 16091:2011 and the accelerated oxidation method according to EN 15751 correlate well;

b) the PetroOXY test according to EN 16091:2011 has a good applicability with an R of about 18 % of the test results.

B.4 RRT 2008-231

B.4.1 Layout

In this RR, 19 laboratories participated. The 23 samples tested are given in Table B.2. Each sample was tested twice.

Table B.2 — Layout of the samples

Sample number	Fuel type
S1	B100
S2	B100
S3	B100
S4	B100
S5	B10
S6	B30
S7	B5
S8	B10
S9	B30
S10	B5
S11	B10
S12	B30
S13	B5
S14	B10
S15	B30
S16	B5
S17	B10
S18	B10
S19	B5
S20	B5
S21	B0
S22	B0
S23	B0

B.4.2 Input data

The input data received from the 19 participating laboratories are given in Table B.3.

Table B.3 — Input data of RRT 2008-231

Input Data

Repeats: 2

\+ = valid data pair
C = cochran outlier (dispersion)
H = Hawkins Outlier (position)
X = missing or withdrawn data

	Sample No	S1			S2			S3			S4			S5			S6			S7			S8		
Lab Code	P21	+	22,63	23	+	53,65	54,57	+	28,2	28,23	+	24,23	24,4	+	77,75	75,53	+	40,7	40,63	+	100,95	100,55	+	124,27	123,98
	P3	+	21,62	21,43	+	51,88	52,85	+	26,55	26,67	+	22,67	22,57	+	80,32	81,6	+	41,42	41,38	+	99,85	100	+	130,27	130,23
	P22	+	22,35	22,58	+	51,82	52,12	+	26,43	26,8	+	23	23,47	+	78,4	78,73	+	40,83	41,07	+	96,3	94,83	+	125,05	125,25
	P23	+	22,1	21,78	+	52,92	53,3	+	27,17	26,98	+	23,25	23,48	+	79,52	79,77	+	41,38	41,38	+	98,57	97,6	+	126,33	126,37
	P25	+	23,73	23,65	+	55	55,25	+	28,95	29,1	+	25,25	25,33	+	81,4	81,77	+	41,5	41,52	+	101,63	101,28	+	126,7	127,57
	P17	+	21,18	20,5	+	48,5	48,38	+	25,37	24,9	+	22,17	21,55	+	76,35	75,92	+	40,3	40,12	+	92,22	91,82	+	120,47	120,87
	P19	+	21,87	21,93	+	52,93	52,83	+	26,68	26,82	+	23,78	23,55	+	78,77	80,07	+	41,38	41,05	+	96,05	97,57	+	126,32	127,58
	P20	+	22,63	23,03	+	54,68	55,77	+	29,5	29,7	+	25,1	25,07	+	84,23	84,22	+	41,62	42,4	+	101,4	103,05	+	138,88	137,08
	P11	+	22,88	23,18	+	54,03	54,55	+	27,45	27,9	+	24,13	24,12	+	82,75	82,55	+	41,88	41,72	+	102,15	102,55	+	129,8	130,38
	P10	+	22,1	22,05	+	52,17	52,6	+	26,55	26,58	+	22,7	22,87	+	78,48	78	+	40,72	40,83	+	97,85	97,3	+	125,58	125,6
	P5	+	22,02	21,98	+	53,08	53,17	+	26,9	26,82	+	23,05	23,38	+	79,55	80,42	+	40,73	40,63	+	99,87	99,75	+	126	127,57
	P18	+	22,13	22,28	+	52,15	52,17	+	26,83	26,82	+	23,03	23,25	+	78,43	78,3	+	40,17	39,95	+	97,43	97,15	+	123,75	123,1
	P8	+	21,15	21,23	+	52,05	52,43	+	26,83	26,87	+	22,3	22,57	+	76,03	77,5	+	39,97	39,77	+	95,58	96	+	125,8	125,43
	P13	+	22,62	23,22	+	52,67	53,13	+	26,75	27,12	+	24,85	24,5	+	81,17	82,02	+	41,38	41,47	+	98,68	99,27	+	130,75	130,07
	P9	+	22,37	22,62	+	51,65	52,07	+	27,38	27,43	+	23,83	24,17	+	81,35	81,68	+	40,78	40,78	+	100,15	100,82	+	128,47	128,7
	P6	+	18,72	19,32	+	46,02	45,8	+	24,08	24,02	+	20,55	20,72	+	75,57	75,57	+	40	41,03	+	89,85	89,67	+	121,85	126,02
	P7	+	22,82	22,73	+	53,93	54,47	+	26,85	27,18	+	23,83	23,9	+	81,15	81,23	+	39,55	39,62	+	100,67	101,9	+	128,58	127,65
	P24	+	22,05	22,95	C	46,6	52,58	+	28,22	28,38	+	24,78	24,62	+	81,4	79,02	+	42,03	42,68	C	93,92	90,38	+	133,52	130,4
	P16	+	19,98	19,93	+	48,33	47,98	+	24,78	24,53	+	21,68	22,03	+	76,4	76,87	H	37,57	37,6	+	93	92,4	+	122,28	119,13

	Sample No	S9			S10			S11			S12			S13			S14			S15			S16		
Lab Code	P21	+	82,68	83,02	+	139,87	143,1	+	85,88	86,03	+	48,58	48,05	+	107,33	105,58	+	79,48	78,52	+	42,35	42,37	+	95,98	95,38
	P3	+	85,6	85,93	+	145,32	138,32	+	88,33	89,05	C	48,17	49,55	+	109,3	108,82	+	81,45	82,55	+	43,45	43,55	+	99,2	103
	P22	+	84,82	84,53	+	140,98	140,58	+	86,62	86,85	+	49,15	49,35	+	105,6	105,42	+	79,58	79,87	+	43,4	43,35	+	94,52	96,9
	P23	+	85,08	85,27	+	142,17	140,57	+	86,85	86,45	+	48,98	49,48	+	105,5	106,18	+	79,48	79,78	+	43,55	43,75	+	96,37	97,15
	P25	+	86	85,98	+	147,47	146,55	+	90,05	90,03	+	49,37	49,43	+	110,43	110,48	+	82,93	83,03	+	43,63	43,52	+	100,38	99,97
	P17	+	81,17	81,5	+	133,93	134,25	+	81,78	82,87	+	48,23	48,1	+	101,65	101,4	+	75,53	76,5	+	42,32	42,28	+	92,53	92,23
	P19	+	84,48	85,03	+	142,75	142,72	+	88	87,47	+	49,67	49,25	+	107,73	107,63	+	80,98	81,03	+	43,45	43,2	+	97,35	97,9
	P20	+	86,35	86,57	+	150,52	149,83	+	88,15	88,55	+	51,5	51,6	+	109,85	112,45	+	85,93	86,38	+	44,85	45,73	+	98,78	99
	P11	+	87,18	86,83	+	147	145,65	+	88,72	89	+	49,62	50,05	+	110,65	109,3	+	82,33	82,28	+	44,15	44,23	+	100,97	101,47
	P10	+	83,6	83,67	+	139,3	139,62	+	85	85,77	+	49,3	49,1	+	102,92	104,77	+	79,47	79,38	+	42,77	43,22	+	97,38	97,77
	P5	+	84,48	84,8	+	141,77	142,18	+	85,1	85,88	+	48,87	48,9	+	106,57	106,83	+	80,1	80,27	+	42,9	42,53	+	97,93	97,7
	P18	+	81,65	82,1	+	136,67	139,08	+	82,63	84,4	+	48,08	47,72	+	104	104,3	+	77,5	78,37	+	42,03	41,83	+	92,1	96,27
	P8	+	83,92	83,58	+	139,45	141,37	+	86,77	85,57	+	47,75	48	+	106,25	104,8	+	78,95	78,92	+	42,37	42,48	+	95,77	96,52
	P13	+	83,37	84,65	+	146,97	146,85	+	88,67	88,82	+	48,9	48,75	+	108,93	109,35	+	82,58	82,03	+	43,98	44,43	+	101,1	100,05
	P9	+	84,02	84,57	+	144,8	144,28	+	85,8	87,18	+	49,03	49,07	+	108,22	107,85	+	80,78	80,6	+	42,93	43,07	+	99,85	99,7
	P6	+	82,03	84,75	+	135,77	140,05	+	86,77	86,53	+	49,72	49,8	+	102,25	102,92	+	75,63	77,43	+	42,92	43	+	89,48	85,73
	P7	+	83,65	83,45	+	145,57	144,65	C	87,42	83,17	+	49,43	49,28	+	108,83	108,96	+	81,77	80,6	+	41,97	42,07	+	101,57	101,2
	P24	+	84,62	86,9	+	135,23	139,18	+	89,4	90,45	+	50,87	51,33	C	96,55	104,87	+	81,95	79,75	+	44,05	44,52	+	99,63	99,88
	P16	H	77,87	76,73	+	138,22	136,35	+	80,47	80,15	+	45,05	45,17	+	101,97	100,52	+	76,53	76,23	+	40	40,3	+	93,4	92,55

	Sample No	S17			S18			S19			S20			S21			S22			S23		
Lab Code	P21	+	92,82	92,9	+	36,18	36,13	+	47,7	47,67	+	48,35	47,63	+	146,8	141,4	+	100,73	109,47	+	68,68	71,12
	P3	+	95,52	92,6	+	37,23	37,18	+	43,83	43,03	+	47,73	48,28	C	148,17	129,42	+	114,92	107,43	+	74,82	73,45
	P22	+	93,25	93,9	+	35,82	34,55	+	46,73	47,22	+	45,2	44,52	+	133,05	133,98	+	92,77	89,47	+	70,53	69,38
	P23	+	94,92	92,73	+	34,52	34,87	+	46,23	45,98	+	42,93	45,55	+	130,23	132,45	+	103,43	102,1	+	70,17	70,35
	P25	+	96,95	96,97	+	37,67	37,38	+	48,35	48,27	+	46,52	46,07	+	153,58	151,62	+	115,07	115,25	+	69,45	69,4

B.4.3 Precision results of the RRT 2008-231

The precision results of each sample are given in Table B.4.

Table B.4 — Precision results of the samples in RRT 2008-231

unit: minutes
Labs: 19
Samples: 23
Repeats: 2

Precision using all 23 samples:
Repeatability r = -0,7787 + 0,0429 * MW [minutes]
Reproducibility R = -2,2473 + 0,1532 * MW [minutes]

Precision without samples 21, 22:
Repeatability r = -0,1429 + 0,0283 * MW [minutes]
Reproducibility R = 1,1749 + 0,0821 * MW [minutes]

Sample No	S1	S2	S3	S4	S5	S6	S7	S8
Labs / valid labs	(19) 19	(19) 18	(19) 19	(19) 19	(19) 19	(19) 18	(19) 18	(19) 19
Mean	22,009	52,247	26,956	23,414	79,468	40,955	97,936	127,043
Repeatability, r (RV)	0,763	1,079	0,476	0,558	2,013	0,774	1,717	3,215
Reproducibility R (RV)	3,274	7,371	4,032	3,605	7,279	2,220	10,943	11,911
df (R)	19	17	18	18	20	19	17	19
AD (RV)	0,74	1,10	0,69	0,16	0,28	0,15	0,47	0,32
R / mean (%)	15	14	15	15	9	5	11	9

Sample No	S9	S10	S11	S12	S13	S14	S15	S16
Labs / valid labs	(19) 18	(19) 19	(19) 18	(19) 18	(19) 18	(19) 19	(19) 19	(19) 19
Mean	84,384	141,814	86,557	49,015	106,543	80,170	43,066	97,228
Repeatability, r (RV)	1,976	4,966	1,673	0,601	2,269	1,751	0,634	3,491
Reproducibility R (RV)	4,690	12,606	7,714	4,094	9,111	7,282	3,239	10,932
df (R)	20	21	18	17	18	19	19	20
AD (RV)	0,16	0,22	0,46	0,78	0,24	0,25	0,29	0,47
R / mean (%)	6	9	9	8	9	9	8	11

Sample No	S17	S18	S19	S20	S21	S22	S23
Labs / valid labs	(19) 19	(19) 18	(19) 19	(19) 18	(19) 18	(19) 19	(19) 18
Mean	93,462	36,788	45,539	47,502	135,236	102,119	70,248
Repeatability, r (RV)	2,451	1,034	1,527	2,760	7,497	8,989	2,402
Reproducibility R (RV)	10,339	4,143	6,972	5,957	37,301	26,011	6,136
df (R)	19	18	19	21	18	20	20
AD (RV)	0,40	0,43	0,39	0,26	0,29	0,47	0,26
R / mean (%)	11	11	15	13	28	25	9

The calculated precision according to EN ISO 4259 ***for all 23 samples is*** as follows:

$$r = -0{,}778\,7 + 0{,}042\,9 \cdot X \quad \text{(B.1)}$$

$$R = -2{,}247\,3 + 0{,}153\,2 \cdot X \quad \text{(B.2)}$$

where

r is the repeatability (the difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material), in min;

R is the reproducibility (the difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material);

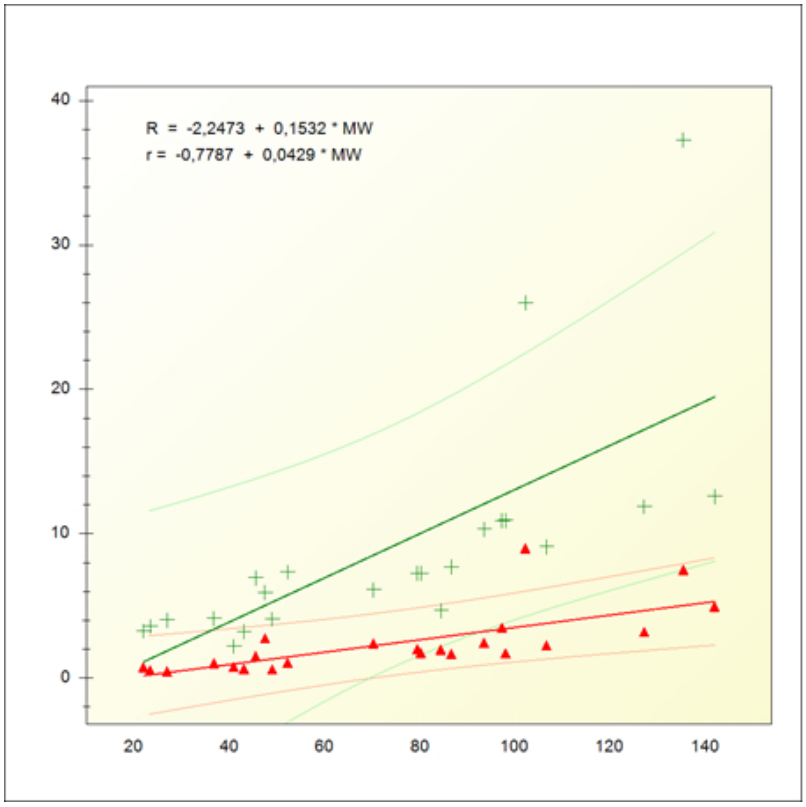
X represents the mean of the two results expressed in min, rounded to the nearest 0,01 min.

The calculated precision according to EN ISO 4259 ***without the samples 21 and 22*** is as follows:

$$r = -0{,}142\,9 + 0{,}028\,3 \cdot X \quad \text{(B.3)}$$

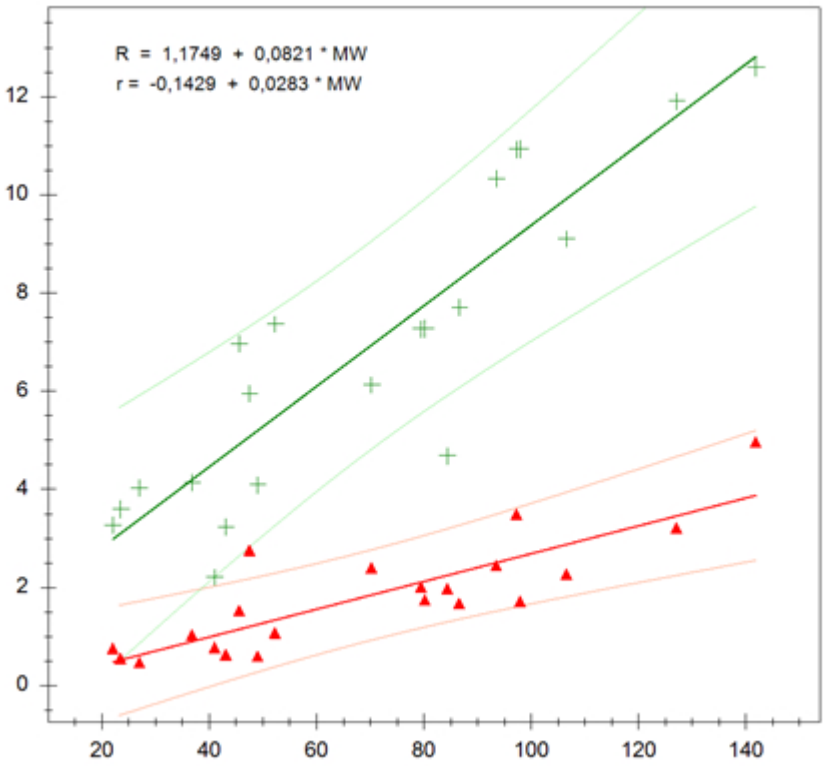
$$R = 1{,}174\,9 + 0{,}082\,1 \cdot X \quad \text{(B.4)}$$

Figures B.2 (samples 21 and 22 included) and B.3 (samples 21 and 22 not included) show a graphical illustration of the above mentioned equations including all precision footprints. The samples 21 and 22 were classified as outliers in RRT 2008-231.



Key
X-axis mean of the two results expressed in min;
Y-axis repeatability, *r* (triangles) respectively the reproducibility, *R* (plusses)

Figure B.2 — Graphical illustration of RRT 2008-231 precision including samples 21 and 22



Key
X-axis mean of the two results expressed in min,
Y-axis repeatability, *r* (triangles) respectively the reproducibility, *R* (plusses)

Figure B.3 — Graphical illustration of RRT 2008-231 precision excluding samples 21 and 22

B.5 RRT 2010-231

B.5.1 Layout

Here, 11 laboratories participated. The samples used to test the influence of EHN are given in Table B.5.

Table B.5 — Layout of the samples

FUEL	EHN 0 mg/kg	EHN 500 mg/kg	EHN 1 000 mg/kg
B01	X	X	X
B02	X	X	X
B03	X	X	X
B04	X	X	X
B05	X	X	X
B7-1	X	X	—
B7-2	X	X	—

B.5.2 Input data and precision results

The input data and precision results for RRT 2010-231 for the samples without EHN are given in Table B.6.

Table B.6 — Input data and the precision results for the samples without EHN

Sample	B01			B02			B03			B04			B05			B7#1			B7#2	
LAB No																				
1	+	58,07	58,15	+	71,98	73,07	+	88,92	89,20	+	129,35	130,90	+	212,50	213,93	+	90,60	92,87	+	129,45	130,33
3	m	66,60	71,08	+	72,82	77,63	C	83,58	99,13	m	100,42	103,77	+	213,35	222,37	m	61,97	65,23	+	143,17	146,05
4	+	61,47	61,50	+	69,25	69,90	+	81,58	81,37	m	110,30	109,00	+	224,40	223,92	+	87,23	86,73	+	132,67	131,65
5	+	58,37	61,85	+	72,15	72,45	+	95,03	95,16	C	139,75	121,22	+	216,78	209,17	+	102,10	101,63	+	149,23	144,57
6	+	60,22	60,30	+	71,28	72,40	+	90,27	88,73	+	144,82	144,20	+	212,30	210,57	+	101,32	101,65	+	146,98	145,15
7	+	60,67	61,30	+	67,13	65,48	+	70,50	70,68	+	123,30	122,78	+	216,02	214,62	+	103,63	103,90	+	150,42	146,77
8	+	59,21	59,33	+	66,50	69,08	+	87,35	82,82	+	141,06	138,12	+	212,77	214,87	+	102,42	101,12	+	155,10	153,83
9	+	60,22	60,03	+	69,68	68,98	+	87,32	87,83	+	144,27	143,32	+	215,73	214,08	+	103,45	101,93	+	152,57	150,53
10	+	59,53	60,47	+	66,62	69,05	+	80,77	82,87	+	118,07	119,45	+	204,02	199,28	m	67,20	67,47	H	105,80	106,67
11	H	57,11	56,45	H	61,78	64,58	H	69,70	62,88	H	70,27	61,97	H	135,08	115,00	H	77,20	50,68	H	78,25	64,83
12	+	58,38	58,08	+	70,78	62,78	+	81,18	81,92	+	134,48	135,27	+	221,50	221,27	m	74,48	75,27	+	147,75	148,65

Sample	B01	B02	B03	B04	B05	B7#1	B7#2
(Labs) valid Labs	(11) 9	(11) 10	(11) 9	(11) 7	(11) 10	(11) 7	(11) 9
Mean	59,842	69,951	84,639	133,528	214,673	98,613	144,715
r (RV)	2,773	7,256	4,006	3,470	9,360	2,793	5,591
R (RV)	3,974	10,107	22,882	34,864	19,980	22,663	27,460
DF (R)	12	14	8	6	11	6	8
AD (RV)	0,36	0,30	0,32	0,25	0,41	0,90	0,65

It should be noted that the laboratory 11 turned out as Hawkins outlier for all samples. In addition, several laboratories had some difficulty with samples B04 and B7-01.

The input data and precision results for the samples for RRT 2010-231 with a content of 500 mg/kg EHN are given in Table B.7.

Table B.7 — Input data and the precision results for samples with a content of 500 mg/kg EHN

Sample	B01			B02			B03			B04			B05			B7#1			B7#2	
Lab No																				
1	+	57,13	57,53	+	56,12	56,93	+	69,82	71,28	+	100,90	101,87	+	146,43	146,45	+	67,52	68,28	+	99,02	100,92
3	+	66,35	68,18	+	62,00	61,08	C	86,30	81,00	+	96,42	95,70	+	142,57	142,82	H	51,75	51,18	H	90,93	91,33
4	+	60,23	60,38	+	57,10	57,53	+	70,53	69,97	+	101,30	102,57	+	152,15	153,55	+	64,93	64,58	+	107,90	107,80
5	+	62,17	59,47	+	58,55	58,32	+	72,33	72,53	+	101,15	102,37	+	146,53	148,13	+	75,32	74,53	+	109,87	107,72
6	+	58,70	58,58	+	58,20	58,42	+	68,58	68,90	+	100,32	101,43	+	144,27	143,80	+	77,45	77,42	+	112,67	112,58
7	+	59,60	59,10	+	55,97	55,95	+	64,85	63,87	+	99,65	99,68	+	144,53	144,40	+	77,73	76,38	+	113,05	112,12
8	+	58,03	58,18	+	56,22	55,82	+	69,80	69,87	+	100,40	100,45	+	146,35	147,97	+	77,35	77,37	+	113,03	114,32
9	+	59,50	58,75	+	58,85	58,68	+	71,05	70,05	+	104,17	100,70	+	146,82	145,10	+	79,15	78,10	+	112,22	110,85
10	+	59,62	60,25	+	55,40	57,20	+	71,08	70,05	+	101,60	100,45	+	147,15	148,38	H	56,62	56,17	H	85,40	85,28
11	H	55,07	55,93	H	53,32	47,38	H	61,82	61,90	H	94,50	88,62	H	134,55	136,27	H	37,10	33,10	H	80,98	74,15
12	+	56,20	56,40	+	51,33	50,18	+	71,15	67,53	+	93,60	93,98	+	143,35	144,98	+	70,22	71,00	+	114,55	115,00

Sample	B01	B02	B03	B04	B05	B7#1	B7#2
(Labs) valid Labs	(11) 10	(11) 10	(11) 9	(11) 10	(11) 10	(11) 8	(11) 8
Mean	59,718	56,993	69,624	99,936	146,287	73,583	110,226
r (RV)	2,454	1,804	3,259	3,098	2,692	1,800	2,929
R (RV)	9,573	8,966	7,366	9,115	9,183	17,320	16,022
DF (R)	10	9	10	10	10	7	7
AD (RV)	0,75	0,52	0,82	0,97	0,45	0,54	0,58

The input data and precision results for the samples for RRT 2010-231 with a content of 1 000 mg/kg EHN are given in Table B.8.

Table B.8 — Input data and the precision results for samples with a content of 1 000 mg/kg EHN

Sample	B01			B02			B03			B04			B05	
Lab No														
1	+	54,40	55,05	+	53,10	53,52	+	64,17	64,47	+	83,80	84,77	+	118,57	119,03
3	+	58,22	57,60	+	53,67	52,50	+	60,78	60,83	H	70,38	69,27	+	118,28	117,43
4	+	58,08	57,60	+	54,10	54,75	+	64,83	64,52	H	78,30	77,43	+	123,58	123,55
5	+	57,67	57,18	+	52,75	53,27	C	65,63	70,37	+	85,93	84,42	+	121,53	121,67
6	+	56,32	56,73	+	53,00	53,67	+	63,07	63,38	+	86,43	88,52	+	117,30	117,47
7	+	58,05	57,68	+	52,83	52,92	+	60,32	61,00	+	88,72	89,23	+	118,50	117,97
8	+	56,65	55,82	+	52,38	52,97	+	65,20	64,95	+	91,38	90,30	+	121,02	121,50
9	+	56,37	57,33	+	53,05	52,77	+	63,52	64,85	+	90,22	90,78	+	119,97	120,17
10	+	57,67	57,67	+	55,83	55,18	+	65,20	65,08	H	76,08	75,58	+	121,52	119,57
11	H	53,47	53,77	H	49,88	49,72	H	58,10	58,47	H	58,37	51,98	H	108,08	108,17
12	+	54,35	54,10	+	51,93	51,38	+	62,50	62,33	+	81,93	82,45	+	115,62	115,55

Sample	B01	B02	B03	B04	B05
(Labs) valid Labs	(11) 10	(11) 10	(11) 9	(11) 7	(11) 10
Mean	56,727	53,279	63,389	87,063	119,490
r (RV)	1,272	1,383	1,220	2,764	1,630
R (RV)	4,284	3,369	5,746	11,577	7,627
DF (R)	10	11	8	6	9
AD (RV)	0,64	0,64	0,50	0,23	0,14

B.5.3 Conclusions

From the input data received, it can be concluded that some samples had shown somewhat "misbehaving" results.

Furthermore an effect of EHN on PetroOXY (EN 16091) and Rancimat (EN 15751) could be observed. This effect depends on EHN level and on the sample but it is not clear what the major effects had been in detail. The effect is more visible for PetroOXY (EN 16091) than for Rancimat (EN 15751) and lead in case of the PetroOXY (EN 16091:2010) to a (maybe systematic) decrease of the result. In case of the Rancimat (EN 15751) the effect is not as visible, and it was assumes that it could be possible that smaller effects could be hidden underneath a somewhat larger dispersion of results. Since the effect could not be quantified until now, a general warning note should be included.

Recognizing the above results, the following proposals were made to limit the measurement range to avoid a bad precision.

— for the Rancimat (EN 15751) the test should not exceed the measurement time of about 50 h to 60 h;

— for the PetroOXY (EN 16091) the test should not exceed about 120 min to 130 min.

B.6 Combined results from RRT 2008-231 and RRT 2010-231

The combined result triplets from RRT 2008-231 and RRT 2010-231 sorted by sample number are given in Table B.9.

Table B.9 — Combined result triplets from RRT 2008-231 and RRT 2010-231 sorted by sample number

RRT	Sample	Mean				RRT	Sample	Mean		
Number	*Number*	*result*	*r*	*R*		*Number*	*Number*	*result*	*r*	*R*
2008-231	S1	22,01	0,76	3,27		2010-231	B1 -0000	59,84	2,77	3,97
2008-231	S2	52,25	1,08	7,37		2010-231	B2 -0000	69,95	7,26	10,11
2008-231	S3	26,96	0,48	4,03		2010-231	B3 -0000	84,64	4,01	22,88
2008-231	S4	23,41	0,56	3,61		2010-231	B4 -0000	133,53	3,47	34,86
2008-231	S5	79,47	2,01	7,28		2010-231	B5 -0000	214,67	9,36	19,98
2008-231	S6	40,95	0,77	2,22		2010-231	B7-1 -0000	98,61	2,79	22,66
2008-231	S7	97,94	1,72	10,94		2010-231	B7-2 -0000	144,72	5,59	27,46
2008-231	S8	127,04	3,22	11,91		2010-231	B1 -0500	59,72	2,45	9,57
2008-231	S9	84,38	1,98	4,69		2010-231	B2 -0500	56,99	1,8	8,97
2008-231	S10	141,81	4,97	12,61		2010-231	B3 -0500	69,62	3,26	7,37
2008-231	S11	86,56	1,67	7,71		2010-231	B4 -0500	99,94	3,1	9,12
2008-231	S12	49,01	0,6	4,09		2010-231	B5 -0500	146,29	2,69	9,18
2008-231	S13	106,54	2,27	9,11		2010-231	B7-1 -0500	73,58	1,8	17,32
2008-231	S14	80,17	1,75	7,28		2010-231	B7-2 -0500	110,23	2,93	16,02
2008-231	S15	43,07	0,63	3,24		2010-231	B1 -1000	56,73	1,27	4,28
2008-231	S16	97,23	3,49	10,93		2010-231	B2 -1000	53,28	1,38	3,37
2008-231	S17	93,46	2,45	10,34		2010-231	B3 -1000	63,39	1,22	5,75
2008-231	S18	36,79	1,03	4,14		2010-231	B4 -1000	87,06	2,76	11,58
2008-231	S19	45,54	1,53	6,97		2010-231	B5 -1000	119,49	1,63	7,63
2008-231	S20	47,5	2,76	5,96						
2008-231	S21	135,24	7,5	37,3						
2008-231	S22	102,12	8,99	26,01						
2008-231	S23	70,25	2,4	6,14						

The combined result triplets from RRT 2008-231 and RRT 2010-231 sorted by mean value are given in Table B.10.

Table B.10 — Combined result triplets from RRT 2008-231 and RRT 2010-231 sorted by mean value

RRT	Sample	Mean			outlier		RRT	Sample	Mean			outlier
Number	Number	result	r	R	Type		Number	Number	result	r	R	type
2008-231	S1	22,01	0,76	3,27			2008-231	S14	80,17	1,75	7,28	
2008-231	S4	23,41	0,56	3,61			2008-231	S9	84,38	1,98	4,69	
2008-231	S3	26,96	0,48	4,03			2010-231	B3 -0000	84,64	4,01	22,88	
2008-231	S18	36,79	1,03	4,14			2008-231	S11	86,56	1,67	7,71	
2008-231	S6	40,95	0,77	2,22			2010-231	B4 -1000	87,06	2,76	11,58	Y
2008-231	S15	43,07	0,63	3,24			2008-231	S17	93,46	2,45	10,34	
2008-231	S19	45,54	1,53	6,97			2008-231	S16	97,23	3,49	10,93	
2008-231	S20	47,50	2,76	5,96			2008-231	S7	97,94	1,72	10,94	
2008-231	S12	49,01	0,60	4,09			2010-231	B7-1 -0000	98,61	2,79	22,66	Y
2008-231	S2	52,25	1,08	7,37			2010-231	B4 -0500	99,94	3,10	9,12	
2010-231	B2 -1000	53,28	1,38	3,37			2008-231	S22	102,12	8,99	26,01	X
2010-231	B1 -1000	56,73	1,27	4,28			2008-231	S13	106,54	2,27	9,11	
2010-231	B2 -0500	56,99	1,80	8,97			2010-231	B7-2 -0500	110,23	2,93	16,02	Y
2010-231	B1 -0500	59,72	2,45	9,57			2010-231	B5 -1000	119,49	1,63	7,63	
2010-231	B1 -0000	59,84	2,77	3,97			2008-231	S8	127,04	3,22	11,91	
2010-231	B3 -1000	63,39	1,22	5,75			2010-231	B4 -0000	133,53	3,47	34,86	Z
2010-231	B3 -0500	69,62	3,26	7,37			2008-231	S21	135,24	7,50	37,30	X,Z
2010-231	B2 -0000	69,95	7,26	10,11			2008-231	S10	141,81	4,97	12,61	Z
2008-231	S23	70,25	2,40	6,14			2010-231	B7-2 -0000	144,72	5,59	27,46	Z
2010-231	B7-1 -0500	73,58	1,80	17,32	Y		2010-231	B5 -0500	146,29	2,69	9,18	Z
2008-231	S5	79,47	2,01	7,28			2010-231	B5 -0000	214,67	9,36	19,98	Z

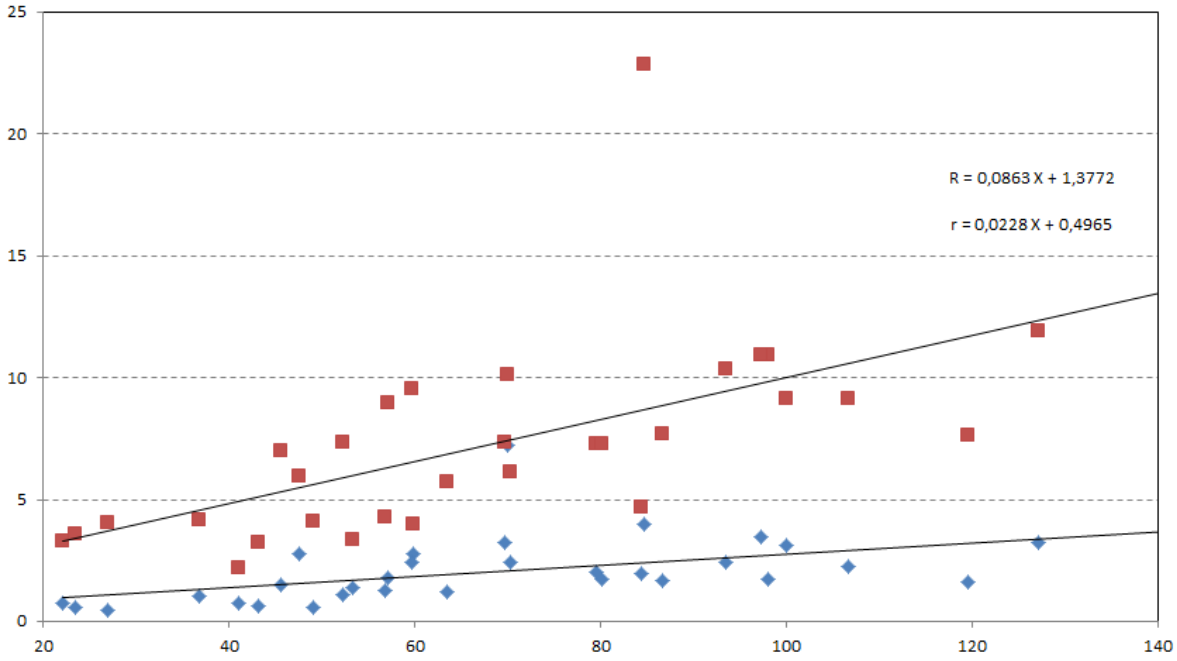
Outlier Type Z: above agreed measurement range (130 minutes)
Outlier Type Y: R much higher compared to meighor samples
Outlier Type X: outlier identified in RRT 2008-231

The precision footprint for the combined RRT exercises is shown in Figure B.4. It is recommended to use these regression functions in the test method.

$$r = 0{,}022\ 8\ X + 0{,}496\ 5 \quad \text{(B.5)}$$

$$R = 0{,}086\ 3\ X + 1{,}277\ 2 \quad \text{(B.6)}$$

NOTE The precision functions are results from regression using the triplets (X, r, R) from RRT 2008-231 and RRT 2010-231 combined.



Key
X-axis mean of the two results expressed in min,
Y-axis repeatability, *r* (diamonds) respectively the reproducibility, *R* (squares)

Figure B.4 — Precision footprints for the combined precision of the PetroOXY test method

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