PetroOXY for Oxidation Stability of Spark-Ignition Fuel (Gasoline)

For testing storage stability of **Gasoline** the standard methods of test are

ASTM D 525 – IP 40 – ISO 7536 etc.

Users of these methods complain the very long test duration.

PetroOXY is a project to establish a new method for storage stability of liquid fuels as well as an automatic instrument under the idea of

- good repeatable results
- a minimum of time with
- best possible user safety
- handling even by less skilled users

Testing time will not exceed the duration of other test methods for releasing fuel from production facility or other storage.

The defined event is : **Oxystab = Elapsed Time from test start till (Max-Pressure - 10 %)**

The **PetroOXY-Instrument** is
- a test chamber receiving a volume of 5 ml of sample, with
- automatic oxygen filling and relief device,
- automatic heating and re-cooling device
- automatic pressure detection system and
- an interface to transfer test data to a PC and
- a display to show the test result independently.

Productivity is more than **ten times better** than ASTM D 525.

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Mini Round Robin with samples obtained from SGS Fuel Technology Centre, Speyer, Germany

The following slides show test results from

ASTM D 525 evaluated by SGS Fuel Technology Center, Speyer, Germany and PetroOXY evaluated with three different instruments and three different users.

SGS also has selected a variety of good and relatively bad fuel samples to enable for a theoretical calculation of a new pass-fail-criterion with the PetroOXY Method.

Slide 1 European Gasoline; 8 Samples
shows
a spreadsheet of the test data and
a graph of these results combined with
a calculation of correlation.

Fuels in lower left quadrant have **mutually** been **rejected**.
Fuels in upper right quadrant have **mutually** been **accepted**.

Pass-fail line of **ASTM D 525** is **360 minutes** ; 6 hours.
Pass-fail line of **PetroOXY** corresponding to this is **38 minutes**.

Correlation is R^2 = 0.899 .

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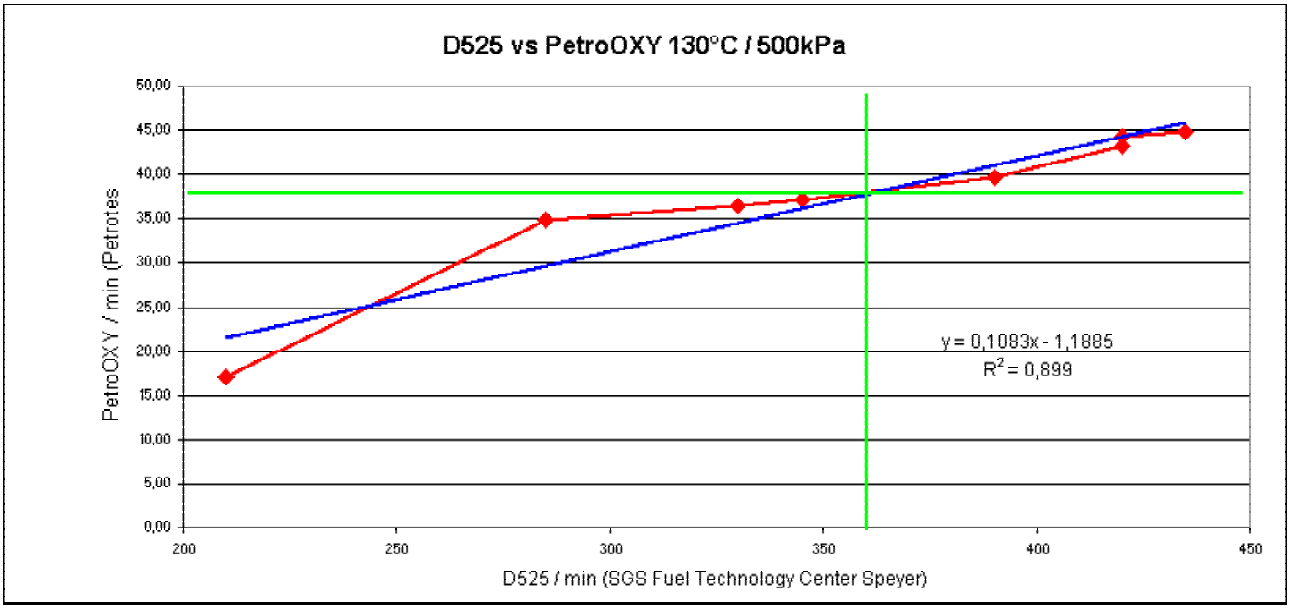
Sample Name	D525	PetroOXY 130°C	Max-Min/MW	STABW/MW
2885	210	17,13	6,23	2,73
1294	285	34,90	9,45	4,22
5658	330	36,51	8,45	3,37
5656	345	37,21	8,87	3,26
5657	390	39,61	7,7	2,55
1644	420	43,20	8,91	3,16
1641	420	44,26	7,98	2,88
1642	435	44,78	6,7	2,52



Slide 2 Precision Statement

shows
a calculation of
Repeatability (r) and **Comparability (R)** .

Except one outlayer both “r” and “R” determined with the PetroOXY are smaller than specified in ASTM D 525.

Calculation was made with ASTM E 691 Adjunct Software package.

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ASTM E11 Quality and Statistical Committee
E691 INTERLABORATORY STUDY of PRECISION PROCEDURE

P R E C I S I O N S T A T E M E N T

PetroOXY Gasoline (European) Mini round robin

Requirements for Determining Precision of Test Method: 1
Carefully examine the data for cases having no data for a particular material within a lab. Unbalanced studies cannot be properly calculated by this program.
(All labs not having the same number of materials)

The number of laboratories, materials, and determinations in this study **DOES NOT** meet the minimum requirements for determinating precision prescribed in ASTM Practice E691:

	This Study	ASTM E691 Minimum
Laboratories	3	6
Materials	10	4
Determinations:	2	2

Precision Statement for Test Method: 1
Precision, characterized by repeatability , Sr, r, and reproducibility, SR, R has been determined for the following materials to be:

Materials	Average	Sr	SR	r	R
485	28.3150	0.6225	1.0019	1.7429	2.8053
1294	34.9033	0.1565	1.8036	0.4383	5.0500
1641	44.2550	0.7586	1.5120	2.1240	4.2335
1642	44.7767	0.3659	1.3676	1.0245	3.8292
1644	43.2033	0.4182	1.6596	1.1710	4.6469
2885	17.1250	0.0372	0.5730	0.1041	1.6043
4169	38.6817	1.0006	6.4187	2.8017	17.9722
5656	37.2117	0.3302	1.4754	0.9245	4.1310
5657	39.6083	0.4528	1.2157	1.2677	3.4040
5658	36.5067	0.0904	1.5057	0.2530	4.2160

This precision statement is provisional. Within five years, additional data will be obtained and processed which does meet the requirements of E691.

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Slide 3 Peruvian Gasoline; 14 Samples

shows
a spreadsheet of the test data and
a graph of these results combined with
a calculation of correlation.

Fuels in lower left quadrant have **mutually** been **rejected**.
Fuels in upper right quadrant have **mutually** been **accepted**.

Fuels in lower right quadrant have been **rejected by PetroOXY**
but **accepted by ASTM D 525**.

Pass-fail line of **ASTM D 525** is **360 minutes** ; 6 hours.
Pass-fail line of **PetroOXY** was also used with **38 minutes**.

Correlation is R^2 = 0.5968 .

Sample name	D 525 / min	PetroOXY / min	Max-Min / MW	STAB / MW
4195	270	20,53	2,76	0,99
4194	270	21,95	2,28	0,73
4198	280	32,24	5,48	1,91
4186	345	27,31	7,26	2,37
4196	345	37,49	4,54	1,63
4187	375	27,19	3,37	1,28
4189	375	30,83	8,49	3,27
4190	405	32,69	13,92	4,97
4191	420	30,93	2,48	1,04
4193	420	31,98	2,55	1,02
4197	420	42,34	7,05	2,42
4200	435	40,16	2,86	0,86
4199	450	42,44	5,03	1,76
4192	460	46,46	2,37	0,78

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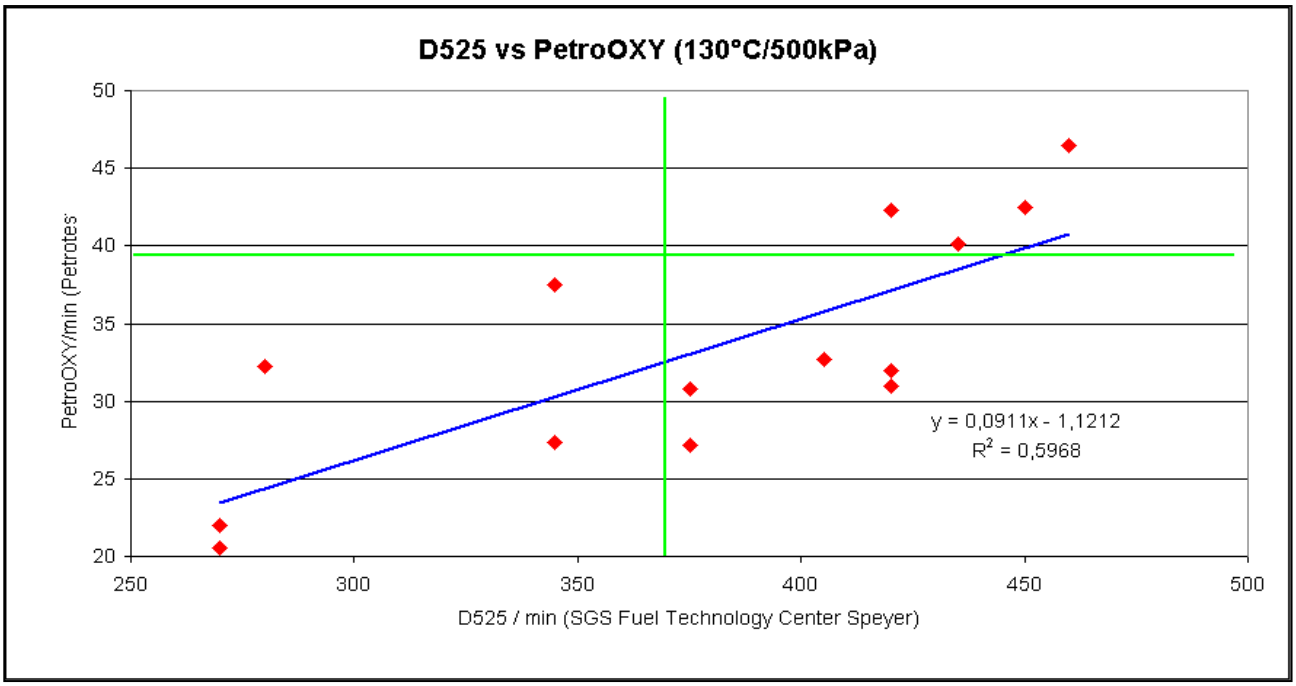

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Slide 4 Precision Statement

shows
a calculation of
Repeatability (r) and **Comparability (R)** .

Both “r” and “R” determined with the PetroOXY
are smaller than specified in ASTM D 525.

Calculation was made with ASTM E 691 Adjunct Software package.

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ASTM E11 Quality and Statistical Committee
E691 INTERLABORATORY STUDY of PRECISION PROCEDURE

P R E C I S I O N S T A T E M E N T

PetroOXY Gasoline (Peru) Mini round robin

Requirements for Determining Precision of Test Method: 1

Carefully examine the data for cases having no data for a particular material within a lab. Unbalanced studies cannot be properly calculated by this program.
(All labs not having the same number of materials)

The number of laboratories, materials, and determinations in this study **DOES NOT** meet the minimum requirements for determinating precision prescribed in ASTM Practice E691:

	This Study	ASTM E691 Minimum
Laboratories	3	6
Materials	14	4
Determinations:	2	2

Precision Statement for Test Method: 1

Precision, characterized by repeatability , Sr, r, and reproducibility, SR, R has been determined for the following materials to be:

Materials	Average	Sr	SR	r	R
4186	27.3150	0.5608	0.7411	1.5704	2.0752
4187	27.1867	0.1637	0.4188	0.4584	1.1728
4189	30.8283	0.2900	1.2237	0.8119	3.4263
4190	32.6883	0.4681	1.9744	1.3107	5.5283
4191	30.9317	0.0706	0.3905	0.1977	1.0935
4192	46.4633	0.1754	0.4332	0.4911	1.2129
4193	31.9767	0.2872	0.3718	0.8042	1.0411
4194	21.9500	0.1274	0.1863	0.3567	0.5215
4195	20.5283	0.0953	0.2453	0.2669	0.6867
4196	37.4867	0.2757	0.7377	0.7719	2.0656
4197	42.3467	0.4715	1.2306	1.3202	3.4456
4198	32.2383	0.4026	0.7267	1.1273	2.0349
4199	42.4450	0.3636	0.8963	1.0180	2.5095
4200	40.1550	0.3742	0.3832	1.0477	1.0729

This precision statement is provisional. Within five years, additional data will be obtained and processed which does meet the requirements of E691.

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Orientation data with LCC Tops and varying additive concentration

In a test program the availability of gasoline samples with differing Oxidation-Stability results was simulated.

Olefins are the most important components in gasoline influencing oxidation stability .
If gasoline has a low olefin content, a high oxidation stability can be expected .
Gasoline in the German market, typically has less than 5% olefin-content.

The idea of our test program was to retrieve a ***"raw"*** gasoline with a high content of olefins and therefore a poor oxidation stability.

German main refineries made the suggestion to use a product named ***LCC top***
(Light cat cracked) with an olefin-content of approx.40%.
In addition to that we received an original antioxidant.

The ***LCC top*** was prepared with different concentration of antioxidant to observe the influence on oxidation stability.

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Slide 5 LCC top
shows
a spreadsheet of the test data obtained by
ASTM D 525 pressure vessel and
PetroOXY at 120°C and 140°C.

PetroOXY vs Standard-OBA (ASTM D 525)

Time in minutes from start until pmax – 10% (mean values)

	D 525 at 100°C	PetroOXY 120°C	PetroOXY 140°C
no additive	113,50	20,73	7,18
125ppm additive	375,50	41,00	11,70
250ppm additive	730,00	67,90	17,22
375ppm additive	843,00	69,68	18,30
500ppm additive	1029,00	81,43	21,50
1000ppm additive	1625,00	92,33	26,43

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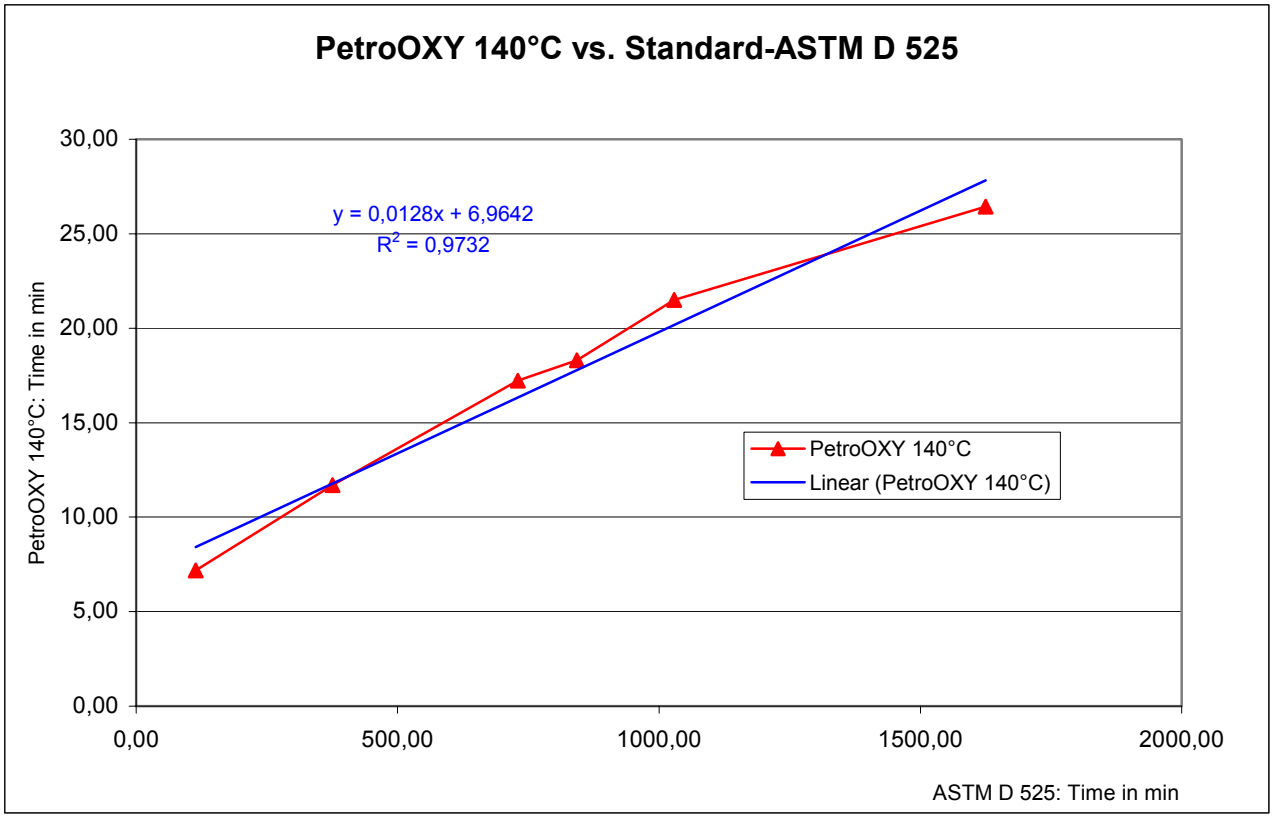
Slide 6 LCC top at 140°C

shows
a graph of the
ASTM D 525 and
PetroOXY test data
Correlation is R^2 = 0.9732668 .



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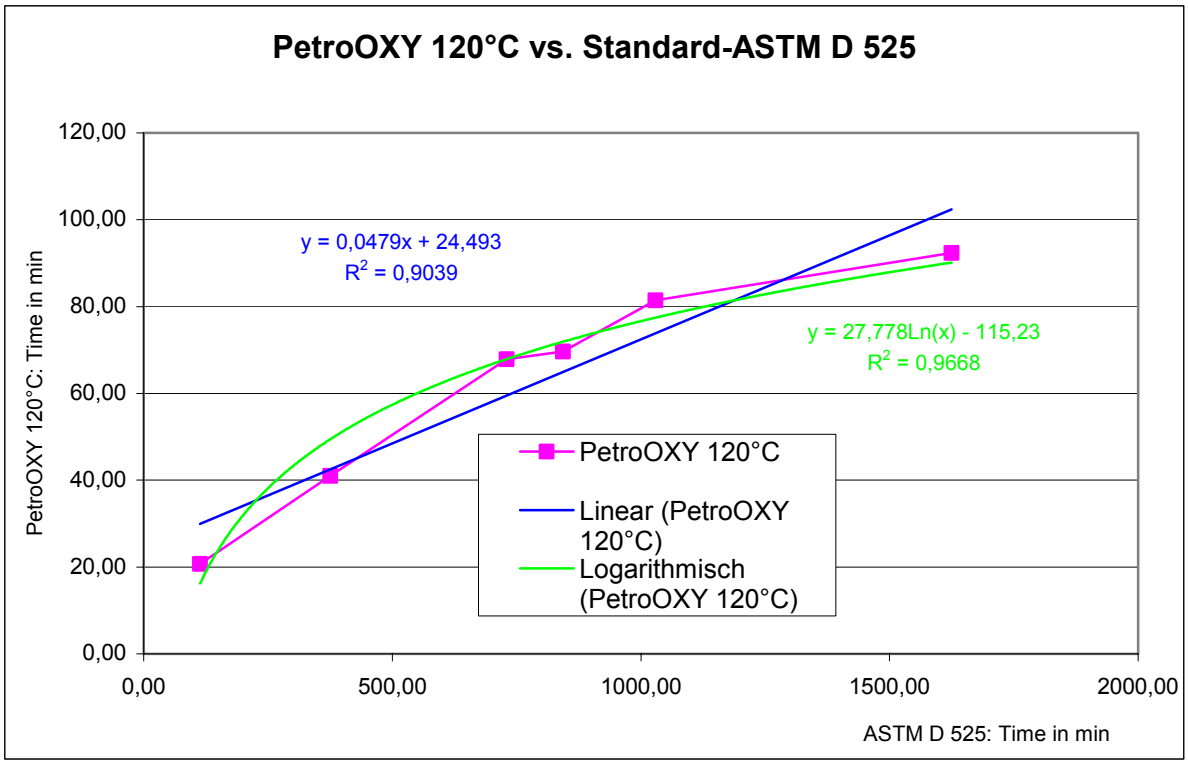
Slide 7 LCC top at 120°C

shows
a graph of the
ASTM D 525 and
PetroOXY test data
Correlation is R^2 = 0.9668 .



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Slide 8 Precision Statement

shows for tests at 140°C
Repeatability (r) = < 4 % and
Comparability (R) = < 5 % .

Both “r” and “R” determined with the PetroOXY
are smaller than specified in ASTM D 525.

Calculation was made with ASTM E 691 Adjunct Software package.

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ASTM E11 Quality and Statistical Committee
E691 INTERLABORATORY STUDY of PRECISION PROCEDURE

P R E C I S I O N S T A T E M E N T

LCC Tops with different amounts of additives at 140C

Requirements for Determining Precision of Test Method: 1

Carefully examine the data for cases having no data for a particular material within a lab. Unbalanced studies cannot be properly calculated by this program.
(All labs not having the same number of materials)

The number of laboratories, materials, and determinations in this study **DOES NOT** meet the minimum requirements for determinating precision prescribed in ASTM Practice E691:

	This Study	ASTM E691 Minimum
Laboratories	2	6
Materials	6	4
Determinations:	2	2

Precision Statement for Test Method: 1

Precision, characterized by repeatability , Sr, r, and reproducibility, SR, R has been determined for the following materials to be:

Materials	Average	Sr	SR	r	R
0 ppm	7.1875	0.0918	0.1718	0.2570	0.4811
125 ppm	11.7050	0.4319	0.5756	1.2092	1.6116
250 ppm	17.2125	0.0391	0.4075	0.1093	1.1411
375 ppm	18.2925	1.1741	1.1741	3.2876	3.2876
500 ppm	21.4975	0.6309	0.8631	1.7665	2.4168
1000 ppm	26.4275	0.8846	1.5173	2.4769	4.2485

This precision statement is provisional. Within five years, additional data will be obtained and processed which does meet the requirements of E691.

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P R E C I S I O N S T A T E M E N T

LCC Tops with different amounts of additives at 120C

Requirements for Determining Precision of Test Method: 1
Carefully examine the data for cases having no data for a particular material within a lab. Unbalanced studies cannot be properly calculated by this program.
(All labs not having the same number of materials)

The number of laboratories, materials, and determinations in this study **DOES NOT** meet the minimum requirements for determinating precision prescribed in ASTM Practice E691:

	This Study	ASTM E691 Minimum
Laboratories	2	6
Materials	6	4
Determinations:	2	2

Precision Statement for Test Method: 1
Precision, characterized by repeatability , Sr, r, and reproducibility, SR, R has been determined for the following materials to be:

Materials	Average	Sr	SR	r	R
0 ppm	20.7350	0.6062	0.7097	1.6973	1.9873
125 ppm	41.0000	2.2701	2.2701	6.3562	6.3562
250 ppm	68.9700	2.6575	2.6575	7.4410	7.4410
375 ppm	69.6725	0.6650	5.6165	1.8620	15.7261
500 ppm	81.4275	0.9794	0.9794	2.7423	2.7423
1000 ppm	92.3375	1.1651	1.5014	3.2624	4.2038

This precision statement is provisional. Within five years, additional data will be obtained and processed which does meet the requirements of E691.

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