#### JSO<del>/FDIS</del>\_4259--5:2023(E)

ISO TC 28/WG 2

Secretariat: NEN

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Petroleum and related products — Precision of measurement methods and results — Part 5: <u>BiasStatistical</u> assessment <u>of agreement</u> between <u>two</u> different <u>measurement</u> methods that claim to measure the same property <del>of a material</del>

## iTeh STANDARD PREV (standards.iteh.ai)

<u>ISO/FDIS 4259-5</u> https://standards.iteh.ai/catalog/standards/sist/48690f70-6793-4182-bc fdis-4259-5

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#### Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documentsdocument should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directiveswww.iso.org/directives).

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#### Introduction

This document explains the statistical methodology for assessing the expected agreement between two standardized test methods that purport to measure the same property of a material. Subsequently, it is investigated whether a linear bias correction can significantly improve the expected agreement. The degree of agreement is expressed as a between-methodmethods reproducibility after a bias correction (if necessary) has been applied.

The method uses numerical results from a set of samples that have been analysed independently using both test methods by different laboratories. The variation associated with each test method result is used for assessing the required bias correction.

Worked<u>Annexes A and B give worked</u> out examples of application of <u>showing how</u> the methodology are given in <u>Annexes A and B</u>is applied.

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### Petroleum and related products — Precision of measurement methods and results — Part 5: <u>BiasStatistical</u> assessment <u>of</u> <u>agreement</u> between <u>two</u> different <u>measurement</u> methods that claim to measure the same property

#### 1 Scope

This document specifies statistical methodology for assessing the expected agreement between two test methods that purport to measure the same property of a material, and <u>for</u> deciding if a simple linear bias correction can further improve the expected agreement.

This document- is applicable for analytical methods which measure quantitative properties of petroleum or petroleum products resulting from a multi-sample-multi-lab study (MSMLS). These types of studies include but are not limited to interlaboratory studies (ILS) meeting the requirements of <u>ISO4259ISO</u> <u>4259</u>-1 or equivalent, and proficiency testing programmes (PTP) meeting the requirements of <u>ISO4259ISO 4259</u>-3 or equivalent. A

The methodology is presented for establishingspecified in this document establishes the limiting value for the difference between two results where each result is obtained by a different operator using different apparatus and two methods X and Y<sub>4</sub> respectively, on identical material, and one. One of the methods (X or Y) has been appropriately bias-corrected to agree with the other in accordance with this practice. This limit is designated as the between-methodmethods reproducibility. This value is expected to be exceeded with a probability of 5 % under the correct and normal operation of both test methods due to random variation.

NOTE Further conditions for application of this methodology are given in 5.1 and 5.2.

#### 2 Normative references

The following documents are referred to in the text in such a way that some <u>or all</u> of their content\* support<u>constitutes</u> requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4259-<u>1</u>, <u>Petroleum and related products</u> — <u>Precision of measurement methods and results</u> — <u>Part 1</u> Determination of precision data in relation to methods of test

ISO 4259--3, Petroleum and related products — Precision of measurement methods and results — Part 3 Monitoring and verification of published precision data in relation to methods of test

ISO 4259-4, Petroleum and related products — Precision of measurement methods and results — Part 4: Use of statistical control charts to validate 'in-statistical-control' status for the execution of a standard test method in a single laboratory

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#### 3 **Terms and definitions**

For the purposes of this document, the terms and definitions in ISO4259ISO 4259-1 and the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

ISO Online browsing platform: available at https://www.iso.org/obphttps://www.iso.org/obp \_

IEC Electropedia: available at https://www.electropedia.org/https://www.electropedia.org/ -

#### 3.1

#### multi-sample-multi-lab study

MSMLS

study in which one or more performance characteristics are determined on the basis of analytical results from multiple samples and multiple laboratories

Note 1 to entry: Under certain conditions, inter laboratory studies and proficiency testing schemes meet this definition of multi-sample-multi-lab study.

#### 3.2 interlaboratory study

#### ILS

study specifically designed to estimate the repeatability and reproducibility of a standard test method achieved at a fixed point in time by multiple laboratories through the statistical analysis of their test results obtained on aliquots prepared from multiple materials

#### 3.3

#### proficiency testing programme

РТР programme designed for the periodic evaluation testing capability of participating laboratories of a standard test method through the statistical analysis of their test results obtained on aliquots prepared from a single batch of homogeneous material

Note 1 to entry: PTP is sometimes referred to as a proficiency testing (PT)-study or an interlaboratory cross checkprogramme (ILCP).

#### 3.4

I

#### between-methodmethods bias correction

quantitative expression of the mathematical correction, when applied to the outcome of either one of two methods claiming to measure the same property, can result in a statistically significant improvement between the expected values of the two test methods claiming to measure the same property

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#### correlation coefficient

P ρ statistical measure of the strength and greation of the relationship between two variables D Note 1 to entry: Values always range between -1 (strong negative relationship) and +1 (strong positive relationship). Values at or close to



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3.82       Sample standard deviation       Formatted: Fort: Bold         estimator of the population standard deviation using the sample mean and sample size       Formatted: Fort: Bold         3.98       Extreme standard deviation is also referred to as standard deviation of the sample.         3.98       Extreme standard deviation of the limiting value that the difference between two single transmission on an identical text sample using different apparatus and applying the two methods. Yang V, respectively, when the methods have been assessed and an appropriate between sub-single transmission. User the methods have been assessed and an appropriate between two methods. Yang V, respectively, when the methods have been assessed and an appropriate between sub-single transmission. Standard deviation is parately to be exactly be assessed and an appropriate between the results for method. Single control in parameter selection and bias-correction model selection.         3.99       Extreme standard deviation of the interindoparatory study. (2:2) in terms of total stand 500000 appropriate between the results for methods after ensembles different apparatus and applying the base correction model selection.         3.91       Extreme study the information content from the interioboratory study. (2:2) in terms of total study to spece between Asian total 300000 appropriate between Asian total 3000000 appropriote between Asian total 3000000 approprise appropri		ng the standard deviation of the distribution of the average statistic obtained from the ampling of a population		
estimator of the population standard deviation using the sample mean and sample size Note 1 to entry: Sample standard deviation is also referred to as standard deviation of the sample. Sage Permethod methods reproducibility Rev. August and the computation of the limiting value that the difference between two single results is expected to be accessed accessed (with a probability of 5% due to random variation, under the correct and normal operation of both test methods, where each result is obtained by different operators on an identical test sample using different apparatus and applying the two methods X and Y, respectively; when the methods have been assessed and an applying the two methods X and Y, respectively; sum of squared results from either method (X or Y) in accordance with this practice 3.1420 Sum of squared results from either method (X or Y) in accordance with this practice 3.1410 Note 1 to entry: Lace is used to quantify the degree of agreement between the results from two test methods after metween methode. <i>Cuence</i> . <i>Method</i> . <i>Classes</i> 10:1 Note 1 to entry: Lace is used as an optimality criterion in parameter selection and bias-correction model selection. 3.1410 Note 1 to entry: Lace is used as an optimality criterion in parameter selection and bias-correction model selection. 3.1410 Note 1 to entry: Lace is used as an optimality criterion in parameter selection and bias-correction model selection. 3.1410 Permated: foot: Ital Permated: foot:	sample standar	d deviation		Formation Forth Park
3-92       Detween methoding terproducibility         K <sub>x</sub> aquantitative expression for the computation of the limiting value that the difference between two single         a comparison of both test methods, where each result is obtained by different operators on an identical test sample using different apparatus and applying the two methods X and Y, respectively; when the methods have been assessed and an appropriate between methoding bias-correction has been asplied to the result from either method (X or Y) in accordance with this practice         3.109       sum of squared residuals       Term STACADARAPPENEV         Sum of squared residuals       Sum of squared residuals         Sufficience in two results that is represented by a different value       Term STACADARAPPENEV         Symbol       Explanation         K_X       reference to the X- and Y-methods, respectively </td <td></td> <td>population standard deviation using the sample mean and sample size</td> <td></td> <td>Formatted: Polit: Bold</td>		population standard deviation using the sample mean and sample size		Formatted: Polit: Bold
between -methods_nethods reproduction of the limiting value that the difference between two single results is concerted to be-acceledezcored with a probability of 5% due to random variation, under the correct and normal operation of both test methods, where each result is obtained by different operators on an identical test sample using different apparatus and applying the two methods X and Y, respectively.       Formatted: For: Bull         3.400       Image: Standard Sta	Note 1 to entry:	Sample standard deviation is also referred to as standard deviation of the sample.		
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		coefficient $\rho \underline{\rho}$ for the X-method and Y-method, respectively		Formatted: Font: 10 pt, Not Italic

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<u>А<del>хі, Ауі</del>Дхі, Д<sub>Уі</sub></u>	absolute deviation of the weighted means of the $i^{\rm th}$ sample results from $\bar{X}$ and $\bar{Y}_{-}$ , respectively	4
Ŷ	predicted Y-method value for a sample by applying the bias correction established from this practice to an actual X-method result for the same sample	4
Ŷ	predicted $i^{\rm th}$ sample Y-method mean, by applying the bias correction established from this practice to its corresponding X-method mean	4
S	number of samples in the multi-lab-multi-sample data set	4
Lxi, Lyi	number of laboratories that returned results on the <i>i</i> <sup>th</sup> sample using the X-method and Y-method, respectively	4
nxij, nyij	number of repeated results on the <i>i</i> <sup>th</sup> sample of <i>j</i> <sup>th</sup> lab using the X- and Y-methods, respectively	4
$R_{X}, R_{Y}$	reproducibility of the X- and Y-methods, respectively	4
Rxi, Ryi	reproducibility of the X- and Y-methods, evaluated at the method X and Y means of the <i>i</i> <sup>th</sup> sample	•
R <sub>XY</sub>	between-methods reproducibility	•
SR,Xi, SR,Yi	reproducibility standard deviation, evaluated at the the <i>i</i> th sample using method X and Y, respectively	•
Sr,Xi, Sr,Yi	repeatability standard deviation, evaluated at the <i>i</i> <sup>th</sup> sample using method X and Y, respectively	
6 <u>8</u>	weighted residual of Y-method mean values predicted from the corresponding X-method mean values, $\hat{Y}_{1}\hat{Y}_{1}$ and mean of Y-method results, $Y_{i}$ on the <i>i</i> <sup>th</sup> sample	
<u>AeAe</u> , xi, <u>AeAe</u> , y <u>i</u>	standard error of the means of the <i>i</i> th sample	à.,
<u>£sr£sr</u> ,p	the weighted sum of squared residuals of the mean results of Y-method and the bias- corrected mean results of the X-method for a given model $p$ where $p = \underline{=} 0, 1q, 1p$ or 2 over all samples $i$	4
<u>-Sst<u>Sst</u>x, <del>Sst<u>S</u>st</del>x</u>	total sum of squares, around the weighted averages $\bar{X}$ and $\bar{Y}$ over all samples $i$	3-4-18
F	test statistic for comparing variances, defined by the quotient of two variances	4
t,	is a student <i>t</i> -value at a specified confidence level and specified degrees of freedom	4
k.	class number of selected bias correction class	4
νχ, νγ	degrees of freedom for reproducibility variances	4
W	weight associated with the difference between (corrected) mean results from the <i>i</i> <sup>th</sup> sample	
a, b	parameter of the bias correction: $\hat{Y} = \underline{a} + bX$	•
h	leverage of sample <i>i</i> in the set of samples	•
Z	natural logarithm of the sample mean, averaged over both methods for sample <i>i</i>	•
Z	overall average of natural logarithm $Z_{\mathbf{k}}$ of all samples	4
t1, t2	ratio for assessing reductions in sums of squares	4
εi	standardized difference between Trant 7 sometimes referred to as error	4
А, В, С	parameters of the quadratic function used for the iterative calculation of the proportional coefficient b for class1bclass1b, and class2class2 correction class	4
D	difference statistic for confirmation of the correlation SEL	4
$\frac{A_i^2}{A_i}, A_i^{2^*}, A_i^2, A_i^{2^*}$	Anderson-Darling test statistic and modified test statistic, respectively	4
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weighted correlation coefficient

#### 5 Procedure overview

#### 5.1 General requirements

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The procedures are intended to be executed by an analyst with sufficient working knowledge of the statistical tools and theories described in the document.

The statistical methodology is based on the premise that a bias correction is not required. In the absence of statistical evidence that a bias correction would improve the expected agreement between the two methods, a bias correction is not made.

If a bias correction is required, then the parsimony principle is followed whereby a simple correction is favoured over a more complex one if the latter does not yield a statistically observable improvement over the former. Failure to adhere to this generally results in a model that is over-fitted and does not perform well in practice.

NOTE-1 The parsimony principle is that the most acceptable explanation of an occurrence, phenomenon, or event is the simplest, involving the fewest entities, assumptions.

The bias corrections of this practice are limited to a constant correction, proportional correction or a linear (proportional + constant) correction.

The bias-correction methods of this practice are method symmetric, in the sense that equivalent corrections are obtained regardless of which method is bias-corrected to match the other.

The methodology described in this document is applicable only if the standard error associated with each mean test result is known or can be calculated and the degrees of freedom associated with all standard errors are at least 30.

This methodology is applied to a data source derived from a MSMLS. The study shall be conducted on at least ten (10) independent materials that span the intersecting scopes of the test methods. The result shall be obtained from at least six (6) laboratories using each method.

The results are obtained on the same comparison set of samples and it is recommended that both test methods are not performed by the same laboratory. If this is the case, care shall be taken to ensure independence of test results, for example by double-blind testing of samples in random order.

This methodology shall not be used on the basis of interim or temporary published precision statements. Interim or temporary statements of accuracy generally lack the magnitude of the amount of data applied and, as a result, insufficient degrees of freedom are available.

Combining multiple data sources is permissible provided the quality requirements for the data set as specified in this document are met.

The test methods used by each laboratory shall be under statistical control, meeting the requirements in ISO4259ISO 4259-4.

This methodology requires data with sufficient resolution to permit variation to be observable in a statistically meaningful manner. Statistically meaningful variation implies that the total number of unique values in a set of data, i.e. the lab results of each sample for each test method, should be sufficiently large. If, in the opinion of the analyst, the number of individual values in the data set is insufficient, the data shall be requested again from the relevant laboratories with sufficient resolution. If the data isare only available with insufficient resolution, this evaluation should not be continued.

In case the data for the procedure originates from an ILS, theall requirements of ISO4259ISO 4259-1 or an equivalent standard, including testing for the existence of extreme samples based on leverage and

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Cook's distance, sample distribution shall be met. Under these conditions and the additional requirements regarding proficiency testing programme (PTP)\_data do not apply.

NOTE-2 Leverage is a measure of how far away the independent variables of an observation are from those of theother observations.

NOTE-3 Cook's distance is an estimate of the influence of a data point-and. It is used within the context of the reference to indicate influential data points that are particularly worth checking for validity.

#### 5.2 Additional requirements for PTP data

#### 5.2.1 General conditions

The statistical calculations are also applicable for this evaluation, provided the results and associated statistics for the test method are obtained from a PTP, which shall meet the requirements of ISO 4259-3 or an equivalent procedure. A characteristic of data derived from such a PTP is that for each sample, a single result is provided by each laboratory for the test method.

The following requirements apply when using PTP data:

- the results shall be obtained from at least ten (10) laboratories using the test method and are equidistantly distributed over the range;
- the leverage of each sample in the data set shall not exceed the limiting value of 0,5 (see 5.2.2<del>),</del>):
- the Anderson-Darling statistics for the tests on normal distribution of lab results per sample-<1,127 shall be used (see 5.2.3);
- the sample standard deviations shall not significantly exceed the published reproducibility standard deviations for at least 80 % of the samples at the 0,05 significance level (see 5.2.4).

#### 5.2.2 Test on existence of extreme samples

The leverage value  $h_i$  for each sample *i* in the data set is examined and may not exceed the limiting value of 0,5. If a value for  $h_i$  of a sample exceeds this limiting value, this sample is characterized as extreme. For each of the two methods, the average of the laboratory results is calculated per sample. Subsequently, each laboratory average per sample is averaged over both test methods.

The leverage value  $h_i$  is defined by Formula-(1):



where

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- h<sub>i</sub> is the leverage of sample *i*,  $i=1 \dots S_i$
- is the total number of samples, is the natural logarithm La) of the sample S

 $Z_i$ over both methods,

Ī is the overall average of all  $Z_i$ .

If one or more samples are characterized as extreme they shall be removed and the procedure should be repeated. The minimum number of remaining samples shall be taken into account. If the minimum requirement of a number of samples can no longer be met, the procedure shall be discontinued.



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#### 5.2.3 Test on distribution of lab results

The distribution of the lab results for each sample are tested for normality by confirming the goodnessof-fit of the normal distribution using the Anderson-Darling statistic per sample.

NOTE-1 The Anderson-Darling test is a statistical test of whether a given sample of data isare drawn from a givenprobability distribution. Within the context of this document, this test is used as <u>a</u> test on normality, with probability distribution parameters (mean and standard deviation) estimated from the sample. See Reference-[7] for further details.

NOTE-2 The critical value of 1,12 is based on a significance level of approximately 1 %, taking into account the effects of rounding of the input data on the resolution.

The test statistic  $\frac{A_i^{2*}}{A_i}A_i^{2*}$  is calculated according to Formula-(2):

$$A_{i}^{2^{*}} = A_{i}^{2} \left( 1 + \frac{0.75}{N_{i}} + \frac{2.25}{N_{i}^{2}} \right)$$
$$----A_{i}^{2^{*}} = A_{i}^{2} \left( 1 + \frac{0.75}{N_{i}} + \frac{2.25}{N_{i}^{2}} \right)$$
(2)

where

here  

$$N_i$$
 is the total number of lab results in the set,  
 $N = \frac{1}{N} \sum_{i=1}^{N} (2i-1) \{ Ln[F(x_i)] + Ln[1-F(x_{N-i+1})] \}_{i}$ 
  
 $A_i^2 = A_i^2 = -N - \frac{1}{N} \sum_{i=1}^{N} (2i-1) \{ Ln[F(x_i)] + Ln[1-F(x_{N-i+1})] \}_{i}$ 
  
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 $F(x_i)$  is the cumulative normal distribution function based on sample average and standard deviation.

 $x_i$  is the data sorted in increasing order,  $x_1 \le x_2 \le x_3 \dots \le x_N \le 59$ .

The distribution of the results is assumed to follow a normal distribution if the corresponding  $\frac{A^{2*}}{A_i}A_i^2$ 

#### 

If this test shows that the distribution of one or more samples does not meet the above criterion, this sample shall be removed. The minimum number of samples for this procedure should be considered. If the minimum requirement of a number of samples can no longer be met, the procedure shall be discontinued.

Data with insufficient resolution due to rounding can overestimate the normality assessment statistics. See 5.1 for resolution provisions.

#### 5.2.4 Comparison of precision

The sample standard deviations  $s_i$  should not significantly exceed the published reproducibility standard deviations  $s_{Ri}$  for at least 80 % of the samples at a significance level of 0,05 using a statistical F-test for the comparison of two variances  $s_i$  and  $s_{Ri}$ .

For any sample *i* where  $s_i$  is numerically larger than  $s_{Ri_k}$  perform the following F-test:—<u>specified in</u> Formula (3):



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