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Nafta in sorodni proizvodi - Natančnost merilnih metod in rezultatov - 4. del: Uporaba grafikonov statističnega nadzora stanja "pod statističnim nadzorom“ za izvajanje standardne preskusne metode v enem laboratoriju

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

Mineralölerzeugnisse - Präzision von Messverfahren und Ergebnissen - Teil 4: Verwendung von Kontrollkarten zur Validierung des Status der statistischen Kontrolle der Durchführung von genormten Prüfverfahren in einem einzelnen Labor

Produits pétroliers -- Fidélité des méthodes de mesure et des résultats -- Partie 4: Utiliser de Cartes Controls Statistique pour valider 'statu d'être en control statistique' par exécution d'une méthode d'essai normalisé en une laboratoire uniquement

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75.180.30	Oprema za merjenje prostornine in merjenje	Volumetric equipment and measurements

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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

Produits pétroliers et connexes — Fidélité des méthodes de mesure et de leurs résultats —

Partie 4: Utilisation de Cartes de Contrôle Statistique pour valider l'état 'sous contrôle statistique' pour l'exécution d'une méthode d'essai normalisée dans un seul laboratoire

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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 documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources.*

A list of all parts in the ISO 4259 series can be found on the ISO website.

Introduction

In the current global business environment, measurement data 'trustworthiness' is a key business driver and an implicit expectation from customers and Regulatory Entities. Data trustworthiness means the data quality meets expectations, and is 'fit-for-use'. Trustworthy data can only be produced by measurement systems that are demonstrated to be stable and are under common cause variation (3.2) only.

This document describes the applications of specific statistical control charts selected from the those that are widely used by the manufacturing sector for the purpose of monitoring and demonstrating the in-statistical-control status of a laboratory in the execution of a standardized test method to produce trustworthy data.

In ISO 4259-2, the requirement for assessment of product quality conformance to specification, is to be interpreted that each laboratory's test result is obtained from a test method that is in statistical control in terms of precision and bias, to be substantiated by in-house SQC charts or other equivalent statistical techniques. While in-house techniques are used by many laboratories for test method quality assurance, standardization on how to establish in-statistical-control is necessary to ensure consistency in application of ISO 4259-2. Addressing the aforementioned necessity is the motivation of this document, which is based on ASTM D6299 [1].

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

1 Scope

This document specifies the work process and methodology for the construction, operation, and maintenance of statistical control charts for the purpose of assessing if a laboratory's execution of a standard test method is in statistical control. By using statistical control charts and following this document the 'in-statistical-control' status is established and validated.

This document explicitly defines 'site precision' conditions as single apparatus, multi-operators, over a long time horizon. It specifies control charts that are most appropriate for ISO/TC 28 test methods where the dominant common cause variation is associated with the long term, multiple operator conditions as described by "site precision" conditions. The control charts specified for determination of in-statistical-control are: Individual (I), Moving Range of 2 (MR2), and either the Exponentially Weighted Moving Average (EWMA) or zone-based run rules (similar to Western Electric (WE) run rules) as sensitivity enhancement strategy to support the I-chart.

The procedures in this document have been primarily designed for numerical results obtained from testing of control samples prepared from a homogenous source of petroleum and related products in a manner that preserves the homogeneity of properties of interest between control samples.

This document applies to properties of interest that are (known to be) stable over time, and for data sets with sufficient resolution to support validation of the assumption that the data distribution can be approximately represented by the Normal (Gaussian) model. Mitigating strategies are suggested for situations where the aforementioned assumption cannot be validated.

2 Normative references

The following documents are referred to in the text in such a way that some of their content support 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 4259-1:2017, *Petroleum and related products — Precision of measurement methods and results — Part 1: Determination of precision data in relation to methods of test*

ISO 4259-2, *Petroleum and related products — Precision of measurement methods and results — Part 2: Interpretation and application of precision data in relation to methods of test*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1 Specific terms and definitions

3.1.1
common cause
factors that contribute to common cause variation.

Note 1 to entry: See Figure 1 for illustration.

3.1.2
common cause variation
variation amongst results collected under site precision conditions from repeated execution of a test method on the same test material attributable to known, unknown, or unknowable factors that are intentionally not or cannot be rigidly controlled as part of the normal and correct execution of all aspects of the test method

3.1.3
special cause variation
variation of a magnitude that exceeds expectation from common cause variation

3.1.4
in-statistical-control
situation wherein the test results produced by the user on control samples are reasonably consistent with expectation over time with common cause variation scattered around a stable expected centre

3.1.5
site precision conditions
conditions under which single test results are obtained in time intervals, separated by at least 8 h, by the testing population in a single laboratory executing the same test method using the same apparatus on test specimens taken at random from the same material over the normal daily operating envelope

3.1.6
control sample
QC sample
specimens taken from a stable and homogeneous material with composition and properties similar to samples normally tested by the laboratory, prepared in a manner that preserves the homogeneity of property of interest between test specimens, stored in a manner that preserves the properties of interest over time, and available in sufficient quantity for repeated long term testing

3.2 Variables and abbreviations

3.2.1
AD
Anderson Darling

3.2.2
$\overline{MR}_{known}$
moving range average associated with the s_{known}

3.2.3
q-q
quantile-quantile

3.2.4

s_{known}

statistically pooled standard deviation obtained using final achieved standard deviations from a group of retired control charts where all the final achieved control chart averages are within 1,5 s_{known}

Note 1 to entry: The range spanned by the final achieved control chart averages are referred to as the working range associated with s_{known} . See concept illustrated in Table 1

Table 1 — Statistically pooled standard deviation concept

material	property (unit)	S_{known}	*df*	working range	$\overline{MR}_{known}$
summer gasoline	vapour pressure (kpa)	0,55	60	49,85 to 50,68	0,62
winter gasoline	vapour pressure (kpa)	0,83	85	104,67 to 105,91	0,93

4 Statistical control in the execution of a standard test method by a laboratory

4.1 General

The execution of a standard test method by a laboratory is in this document considered as the execution of a series of inter-connected work processes. Each work process is subject to variation caused by known, unknown, or sometimes unknowable causes that are inherent in a process over long time horizon such that every outcome of the process is affected. These causes are referred to as common causes. The effect on the final process outcome due to common causes are referred to as common cause variation.

Common causes for variation can be grouped into 5 categories (environment, operator, equipment, procedure and reagent material) using a technique known as a Fishbone diagram. Due to common cause variation, repeat execution of the same test method on the same material over a long time horizon will yield results that are not numerically identical. This effect is illustrated in Figure 1.

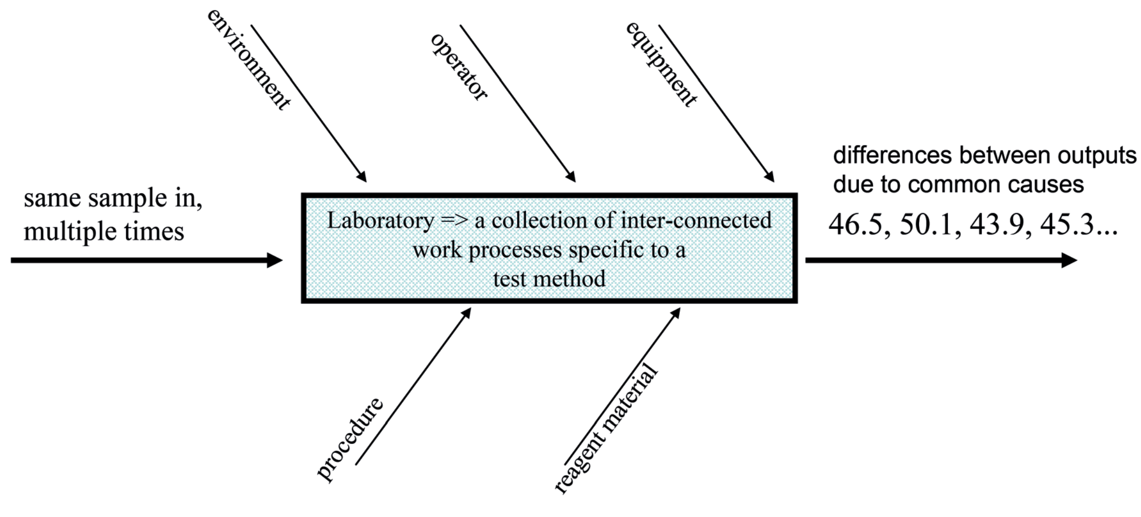

Figure 1 — Fishbone diagram representation of common cause variation in the execution of a test method

The complete process associated with the execution of the specific test method is said to be 'in statistical control' if the process outcomes (test results) from repeated analysis of control (QC) samples prepared from the same material are reasonably consistent with expectation over time; with random variation scattered around a stable centre due to common causes only.

To determine 'in statistical control', a multi-step and integrated work process involving use of statistical control charts and a quality control (QC) material is required.

NOTE For simplicity, the term 'statistical' will be omitted and simply referred to as control charts for the remainder of the document.

4.2 Control Chart description

4.2.1 General

Control Charts appropriate for most petroleum industry test methods that yield numeric results are the Individual (I) chart, and the Moving Range of 2 (MR_2) chart.

NOTE 1 The I-chart is also known as the X-chart, or, the Shewhart chart, named after its inventor, Dr Walter A. Shewhart [2].

NOTE 2 For simplicity, the MR_2 chart is referred to as the Moving Range (MR) chart throughout the rest of the document.

4.2.2 I- and MR-charts

The I-chart is a graphic display of individual control sample test results (X) collected under site precision conditions, plotted in chronological order, overlaid with a centre line, lower and upper decision limits that require action if exceeded. These limits are herein referred to as I-chart lower and upper control limits (LCL_X, UCL_X). The primary purpose of the I-chart is to monitor process centre stability over time.

NOTE Since the primary interest is to monitor the stability and common cause variation of the test process under site precision conditions over a long time horizon, replicate analysis obtained under repeatability conditions (see ISO 4259-1) does not contribute towards this objective, as the variation due to common causes of interest is not contained in replicate results collected under repeatability conditions.

The MR-chart is the successive difference (with no arithmetic sign) of two individual results in the I-chart, plotted in chronological order, also overlaid with a centre line and an upper decision limit for action, herein referred to as MR-chart upper control limit (UCL_MR). The primary purpose of the MR-chart is to monitor common cause variation stability between successive control sample results over time.

The decision limits for action for both charts as well as the conditions requiring action for the strategies in 4.2.3 are based on a very low theoretical probability (<0,3 %) of “action required” decision for a process that is in-statistical-control, using the Normal distribution (I-chart) and W distribution (MR-chart) as the reference statistical models. Hence, these limits represent the expectation limits for the process outcome if it is in-statistical-control.

4.2.3 I-chart sensitivity enhancement strategy

As a direct consequence of setting the action limits for the I-chart based on a low probability of exceedance for a process that is in statistical control, these limits are not sensitive to detection of small changes in the process centre. It is therefore necessary to support I-chart with additional sensitivity enhancement strategies to overcome this shortcoming.

This document requires use of one of the following strategies in conjunction with the I-chart:

1) Strategy 1: Zone-based Run Rules. Action is required if any of the following Run Rule conditions is present. For definitions of zones see 4.3.2 later in this document. These rules are similar to the Western Electric (WE) Run Rules (named after the company that invented them [3]):

   - two out of three consecutive individual results in Zone A on one side of the centre line
   - four out of five consecutive individual results beyond Zone C on one side of the centre line
   - nine consecutive individual results on one side of the centre line

or,

2) Strategy 2: the Exponentially Weighted Moving Average (EWMA). Action is required if either one of the following conditions occur:

   - one or more exceedance of the EWMA action limits
   - nine consecutive individual results on the same side of the centre line (above or below).

The EWMA is a 'time-weighted moving average' calculated using all data points up to the most current one, the weighting of each datum reduced with age exponentially. The rate of this weight decay is controlled by λ. It is re-calculated with the arrival of each new datum, and is judged against its own action limits (herein referred to as the EWMA-action limits). An EWMA with $\lambda = 0.4$ has similar detection power as strategy 1.

NOTE Use of the EWMA with $\lambda = 0{,}4$ as an enhancement strategy was developed by Dr J. Stuart Hunter [4].

Use of Strategy 2 is recommended due to the ease of implementation and lower expected false alarm rate than Strategy 1.

4.2.4 In statistical control conditions

The complete process associated with the execution of a test method execution is deemed to be in statistical control if all of the following conditions are met:

1) all individual control sample results are within the I-chart action limits,
2) less than five (5) out of 12 (12) successive MR results exceed MR-chart action limit, and
3) no action required for the sensitivity enhancement chosen (strategy 1 or 2).

4.3 Control Chart Work Process

4.3.1 General

To determine if the complete process associated with the execution of a test method is 'in statistical control', a two-stage multi-step work process involving use of control charts and QC material is specified by this document.

Stage 1 comprises visual and statistical assessment of initial test results for a new batch of QC material plotted in a chronological order (known as a run chart). This is followed by construction of the I-chart and MR-chart using these results by overlaying the mean and the action limits onto the respective charts. The action limits represent the boundaries within which the current and future test results and MR for this QC material are expected to lie, on the assumption that the process is in statistical control and the QC material remains unchanged. The control charts (I and MR)) constructed in Stage 1 are deployed for Stage 2 if all in-statistical-control conditions (4.2.4) are met.

Stage 2 comprises of two modes, Operation and Maintenance. Under 'Operation', future test results (for the QC material tested in Stage 1) as they arrive in chronological order are compared against the established action limits and chosen enhancement strategy in Stage 1. Under 'Maintenance', the statistics used in the computation of the control chart action limits from Stage 1 are re-assessed periodically using newly accrued in-statistical-control results and updated as appropriate.

For this practice, the root-mean-square technique is used to compute the sample standard deviation statistic, *s*.