

STN	Kvalita vody Kalibrácia a hodnotenie analytických metód Časť 1: Lineárna kalibračná funkcia	STN ISO 8466-1 75 7031
------------	------------------------------------------------------------------------------------------------------------------------	------------------------------------------------

Water quality
Calibration and evaluation of analytical methods
Part 1: Linear calibration function

Qualité de l'eau
Étalonnage et évaluation des méthodes d'analyse
Partie 1: Fonction linéaire d'étalonnage

Táto slovenská technická norma obsahuje anglickú verziu medzinárodnej normy ISO 8466-1: 2021 a má postavenie oficiálnej verzie.

This Slovak standard includes the English version of the International standard ISO 8466-1: 2021 and has the status of the official version.

Nahradenie predchádzajúcich slovenských technických noriem

Táto slovenská technická norma nahrádza STN ISO 8466-1 zo septembra 1995 v celom rozsahu.



135452

Anotácia

Kalibrácia je nevyhnutnou podmienkou kvantifikácie analytov fyzikálno-chemickými a chemickými metódami. Vo väčšine prípadov sa používa jednoduchá lineárna regresia, pretože mnohé metódy merania vykazujú lineárny vzťah medzi indikovanou hodnotou a obsahom vzorky.

Od vydania ISO 8466-1 v roku 1990 sa v oblasti inštrumentálnej analýzy dosiahol obrovský pokrok, čo spôsobilo, že sa vyvinuli rozličné kalibračné stratégie s cieľom čo najlepšieho využitia prístrojového vybavenia. Kalibračný rozsah mnohých analytických metód bol obmedzený na maximálne jeden rád teoretickou štatistickou požiadavkou použiť jednoduchú lineárnu regresiu iba vtedy, ak existuje homogenita rozptylov vo zvolenom pracovnom rozsahu. Kvôli odhadu neistoty merania výpočtom intervalu spoľahlivosti v ISO 8466-1:1990 bolo potrebné splniť požadovanú homogenitu rozptylov. Medzitým boli zavedené iné metódy na odhad neistoty merania, ktoré sú nezávislé od kalibrácie (napríklad ISO 11352).

Kalibrácia sa vykonáva vždy v dvoch krokoch. Prvý krok zahŕňa určenie lineárneho rozsahu, druhý krok je výpočet kalibračnej funkcie. Kalibračné stratégie opísané v tomto dokumente umožňujú analytikovi individuálne definovať kalibračný program podľa špecifikovaných požiadaviek. Metóda opísaná v ISO 8466-1:1990 zostáva súčasťou informatívnej prílohy, pretože môže byť stále užitočná na špecifické účely (napríklad pri validácii metódy).

Národný predhovor

Normatívne referenčné dokumenty

Táto slovenská technická norma neobsahuje normatívne odkazy.

Vypracovanie slovenskej technickej normy

Spracovateľ: Úrad pre normalizáciu, metrológiu a skúšobníctvo SR, Bratislava

Technická komisia: TK 27 Kvalita a ochrana vody

Contents

Page

Foreword.....	v
Introduction.....	vi
1 Scope.....	1
2 Normative references.....	1
3 Terms and definitions.....	1
4 Symbols.....	2
5 Determination of the linear working range and establishment of the calibration range.....	4
5.1 General.....	4
5.2 Preliminary choice of working range.....	4
5.3 Estimation of the linear working range.....	4
5.3.1 General.....	4
5.3.2 Visual testing of measurement data — Testing using the x/y -diagram.....	5
5.3.3 Estimation of the linear range by calculating the point-to-point slope.....	5
6 Calibration strategies.....	6
6.1 General.....	6
6.2 Calculation of the calibration function.....	8
6.3 Calibration of the measuring method using an external standard, including determination of the recovery rate of the analyte.....	9
6.3.1 General.....	9
6.3.2 Establishing the calibration function.....	9
6.3.3 Determination of the recovery rate.....	10
6.3.4 Calculation of results.....	10
6.4 Calibration of the measuring method using an internal standard, including determination of the recovery rate of the internal standard.....	11
6.4.1 General.....	11
6.4.2 Establishing the calibration function.....	11
6.4.3 Determination of the recovery rate.....	11
6.4.4 Calculation of results.....	12
6.5 Calibration of the total procedure using an external standard.....	12
6.5.1 General.....	12
6.5.2 Establishing the calibration function.....	12
6.5.3 Calculation of results.....	13
6.6 Calibration of the total procedure using an internal standard.....	13
6.6.1 General.....	13
6.6.2 Establishing the calibration function.....	13
6.6.3 Calculation of results.....	14
6.7 Standard addition.....	14
6.7.1 General.....	14
6.7.2 Procedure.....	14
6.7.3 Calculation of results.....	15
7 Strategies for testing the validity of calibration.....	16
7.1 General.....	16
7.2 Testing by means of a control solution or control sample.....	16
7.3 Testing the slope of the calibration line.....	16
Annex A (informative) Goodness-of-fit test according to Mandel, standard deviation of the procedure, variation coefficient of the procedure and confidence interval.....	17
Annex B (informative) Examples of linearity testing.....	20
Annex C (normative) Examination of the linear working range using the empirical test of curvature.....	32

Annex D (informative) Weighted regression — Weighting $1/x$	39
Bibliography	41

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 of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This second edition cancels and replaces the first edition (ISO 8466-1:1990), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the title has been modified;
- the scope of the document is the calibration for routine analysis;
- calculation of performance characteristics has been moved to the informative [Annex A](#);
- the calibration range has been extended to several decade orders of magnitudes;
- the verification of the homogeneity of variances has been deleted;
- the linearity test has been modified;
- various calibration strategies have been described;
- the document has been editorially revised.

A list of all parts in the ISO 8466 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Calibration is a prerequisite for the quantification of analytes by means of physicochemical and chemical methods. In most cases, simple linear regression is applied because many measuring methods show a linear relationship between the indicated value and the sample content.

Since the publication of ISO 8466-1 in 1990, a huge progress has been made in the field of instrumental analysis, a consequence of which is that various calibration strategies have been developed in order to make best use of the equipment. The calibration range of many analytical methods was constrained to a maximum of one order of magnitude by the theoretical statistical requirement to only apply simple linear regression if homogeneity of variances exists across the selected working range. Due to the estimation of measurement uncertainty by calculation of the confidence interval in ISO 8466-1:1990, it had been necessary to conform to the required homogeneity of variances. Meanwhile, other methods for the estimation of measurement uncertainty that are independent of calibration have been established (e.g. ISO 11352).

Calibration is always done in two steps. The first step comprises the determination of the linear range, the second step is the calculation of the calibration function. The calibration strategies that are described in this document enable the analyst to individually define the calibration effort according to specified requirements. The method that is described in ISO 8466-1:1990 remains part of the informative annex since it can still be useful for specific purposes (e.g. method validation).

Water quality — Calibration and evaluation of analytical methods —

Part 1: Linear calibration function

1 Scope

This document specifies various calibration strategies for physicochemical and chemical analytical methods and specifies the calculation of analytical results.

It defines the general context for linear calibration so that individual standards dealing with analytical methods for the examination of water quality can make reference to it.

2 Normative references

There are no normative references in this document.

koniec náhľadu – text ďalej pokračuje v platenej verzii STN