
**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



This document is a preview generated by EUS



COPYRIGHT PROTECTED DOCUMENT

© ISO 2021

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

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

This document is a preview generated by EVS

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.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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 <https://www.electropedia.org/>

3.1

working range

interval, determined by calibration, between the highest and the lowest content, where the lowest possible limit of the working range is the limit of quantification of the analytical method

3.2

one-point calibration

calibration between the origin and the indicated value corresponding to the content in the *calibration sample* (3.8)

3.3

two-point calibration

calibration using two *calibration samples* (3.8) with different contents at the upper and at the lower working range limit

3.4

indicated value

y

quantity value provided by a measuring instrument or a measuring system

Note 1 to entry: In accordance with definition 4.1 “indication” of ISO/IEC Guide 99[Z].