
**Gas analysis — Comparison methods
for the determination of the
composition of gas mixtures based on
one- and two-point calibration**

*Analyse des gaz — Méthodes de comparaison pour la détermination
de la composition des mélanges de gaz basées sur un ou deux points
d'étalonnage*

Sample Document

get full document from standards.iteh.ai



Sample Document

get full document from standards.iteh.ai



COPYRIGHT PROTECTED DOCUMENT

© ISO 2017, Published in Switzerland

All rights reserved. Unless otherwise specified, 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
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Symbols	2
5 Abbreviated terms	2
6 Principle	3
6.1 General requirements	3
6.2 Calibration methods	4
7 Main procedure	4
7.1 Pre-requisites	4
7.2 Sequence of operations (overview)	4
7.3 Calibration and measurement designs	5
7.3.1 General	5
7.3.2 Single-point exact-match calibration (SPEM)	6
7.3.3 Single-point through origin calibration (SPO)	6
7.3.4 Two-point calibration with a blank (TPB)	7
7.3.5 Two-point calibration with bracketing (TPC)	7
7.3.6 Multipoint calibration (MPC)	8
8 Performance evaluation of the measuring system	8
8.1 General	8
8.2 Sources of performance evaluation data and alternative approach	11
8.2.1 Performance evaluation other than in-house	11
8.2.2 Alternative for performance evaluation	11
9 Quality assurance measures	11
9.1 Validation of the assumptions made	11
9.2 Drift/stability control of the measuring system	11
10 Report of results	12
10.1 Calibration gas certificates	12
10.2 Report of analysis	12
Annex A (normative) System-stability check required when using exact-match and bracketing designs	13
Annex B (normative) Statistics and sensitivity coefficients used in Clause 7	15
Annex C (normative) Alternative approach to assessing the nonlinearity contribution	17
Annex D (informative) Worked-out example	19
Annex E (informative) Using the designs without preceding system performance evaluation	22
Bibliography	24

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 158, *Analysis of gases*.

Introduction

Whereas the comparison methods described in ISO 6143 based on multipoint calibration are in principle suited for all applications in gas analysis for determining the composition of calibration gas mixtures, in many cases, simpler calibration methods can be used. These methods typically require a smaller number of calibration gas mixtures with a traceable composition.

One- and two-point calibration of instruments is widely used in the gas industry and in national metrology institutes. They often constitute fair compromise between costs and efforts on one hand, and accuracy on the other. These simpler methods require validation to confirm that the conditions of use are appropriate.

It is the intention of this document to set up and describe comparison methods and data evaluation techniques based on one- and two-point calibration. The applicable conditions and limitations of the methods are derived from the analytical requirements and are clearly specified.

This document describes the assessment, calculation and expression of measurement uncertainty arising from significant contributors in the different comparison methods.

Sample Document

get full document from standards.iteh.ai

Sample Document

get full document from standards.iteh.ai

Gas analysis — Comparison methods for the determination of the composition of gas mixtures based on one- and two-point calibration

1 Scope

This document provides methods for

- calibrating an instrument with one or two calibration gas mixtures,
- determining the composition of a gas sample, and
- evaluating the uncertainty of the composition of the gas sample in relation to the uncertainty of the composition of the calibration gases used and the contribution of the measurement process.

This document sets requirements to, and acceptance criteria for, the utilization of different measurement calibration designs with a limited (i.e. minimum) number of calibration gas mixtures used in calibration.

The methods in this document are described for amount-of-substance fractions, but are also applicable for other composition quantities (such as mass fractions, volume fractions or concentrations).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes 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 6141, *Gas analysis — Contents of certificates for calibration gas mixtures*

ISO 6143, *Gas analysis — Comparison methods for determining and checking the composition of calibration gas mixtures*

ISO 7504, *Gas analysis — Vocabulary*

ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

ISO/IEC Guide 99, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7504, ISO/IEC Guide 98-3 and ISO/IEC Guide 99 apply.

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

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Symbols

α_j	coefficients of the best available calibration function f ($j = 0, 1, \dots, N$) with $N \leq 3$ as determined in the performance evaluation procedure, with N being the order of the polynomial
b_j	parameters of the simplified analysis function g ($j = 0, 1$)
β_j	parameters of the best-available analysis function g ($j = 0, 1, \dots, N$) with $N \leq 3$ as determined in the performance evaluation procedure, with N being the order of the polynomial
f	calibration function, $y = f(x)$
g	analysis function, $x = g(y)$
k	coverage factor
m	number of replicate measurements taken
N	order of the polynomial
R	range of amount-of-substance fractions within which the selected method of calibration is applied to samples of unknown composition (hereinafter: unknown samples)
$U(q)$	expanded uncertainty associated with the quantity, q , $U(q) = k \times u(q)$
$u(q)$	standard uncertainty associated with the quantity, q
$u(\Delta)$	uncertainty contribution due to zero-deviations and analyser nonlinearity
$u^2(q)$	variance associated with the quantity, q
x	amount fraction
(x_i, y_i)	calibration point(s) ($i = 1, 2, \dots, n$), with n being its number
$y(l)$	instrumental response, with l indicating the replicate number
$\bar{y}$	mean replicated instrumental responses
Γ	goodness of fit; a measure of local compatibility of a calibration point with the calculated calibration or analysis function
Δ	bias due to zero-deviation and analyser nonlinearity
δ	bias
$\bar{\delta}$	average bias

5 Abbreviated terms

b	blank
GLS	generalized least squares
MPC	multipoint calibration

Re	measurement of the matching reference
r	subscript denoting a calibration gas mixture
r1, r2	calibration gas mixtures bracketing the composition of the unknown sample
S	unknown gas sample
s	subscript denoting an unknown gas sample
SSD	sum of weighted squared deviations
SPeM	single-point exact-match calibration
SPO	single-point through origin calibration
TPB	two-point calibration using a single point and a blank
TPC	two-point calibration

6 Principle

6.1 General requirements

The composition of a gas mixture is determined by separate measurement of the amount fraction of every specified component. "Separate" implies that the response obtained for the component of interest is treated independently of any other response obtained for any other component. It does not impede obtaining several responses for several components in the same instrument run. Therefore, the procedure for determining the amount fraction of only one specified component is described. Possible interferences of other components on the measurement of the component of interest shall be avoided or corrected for separately. This subject is not further addressed in this document.

The procedure for determining the amount fraction x of a specified component X in a sample of a gas mixture, or in a series of such samples, is performed in a sequence of steps as summarized below.

- a) Specify the analytical range of interest, R , i.e. the range of the amount fraction x to be determined, and the acceptable or required (if applicable) uncertainty level.
- b) Specify the analytical method and the measuring system to be used:
 - 1) calibration range (to encompass the analytical range);
 - 2) composition, including uncertainty, of the calibration gas mixtures;
 - 3) parameters of the analytical method;
 - 4) measuring conditions;
 - 5) number and sequence of calibration measurements (see [8.1](#)).
- c) Select, from the list given in [6.2](#), an approach for calibration and determining the amount fractions of components in unknown samples in the regular, i.e. daily mode when one- and two-point based analysis functions are used.
- d) For the relevant ranges of responses and component amount fractions, determine the uncertainty level of the prospective results based on the analysis function. If the result is acceptable, performance evaluation was successful.

For the calculation of the measurement uncertainty of results obtained in the regular calibration mode, an estimate $u(\Delta)$ for the contribution of possible nonlinearity of the system is needed. This estimate is calculated in a performance evaluation of the system according to [Clause 8](#) and [Annex C](#).