INTERNATIONAL STANDARD

ISO 9169

First edition
1994-11-15

Air quality — Determination of performance characteristics of measurement methods

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Qualité de l'air — Détermination des caractéristiques de performance des méthodes de mesurage

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Reference number
ISO 9169:1994(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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International Standard ISO 9169 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 4, *General aspects*.

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Annexes A, B and C form an integral part of this International Standard. Annex D is for information only.

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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Air quality — Determination of performance characteristics of measurement methods

1 Scope

This International Standard specifies procedures to quantify the following performance characteristics of air quality measurement methods defined in ISO 6879[1]: bias (in part only), calibration function and linearity, instability, lower detection limit, period of unattended operation, selectivity, sensitivity, upper limit of measurement.

The procedures given are applicable only to air quality measurement methods with linear[2] continuous calibration functions, the output variable of which is a defined time average. Additionally, replicate values belonging to the same input state are assumed to be normally distributed. Components needed to transform the primary measurement method output into the time averages desired are regarded as integral parts of this measurement method.

For measurement method stability surveillance under routine measurement conditions, it may suffice to check the essential performance characteristics using simplified tests, the degree of simplification acceptable being dependent on the knowledge of the invariance properties of the performance characteristics previously gained by the procedures presented here.

There is no fundamental difference between the instrumental (automatic) and the manual (e.g. wet-chemical) procedures as long as the measured value is an average representative for the predefined time interval. Therefore, the procedures given are applicable to both. Furthermore, they are applicable to measurement methods for ambient air, indoor air, workplace air, and emissions.

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2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3534-1:1993, *Statistics — Vocabulary and symbols — Part 1: Probability and general statistical terms.*

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

ISO 6879:—[3], *Air quality — Performance characteristics and related concepts for air quality measurement methods.*

1) The definition of method in ISO 6879:1983, 4.2.1.9 implies a specific instrumental setup.

2) This linearity may be enforced by a certain amount of postprocessing of the primary output variable.

3) To be published. (Revision of ISO 6879:1983)

3 Definitions

For the purposes of this International Standard the following definitions apply:

NOTE 1 The term *measuring system* used in the context of this International Standard does not constitute a new definition, as compared to the basic terms given in ISO 6879; it merely indicates a tangible realization of a measuring procedure.

3.1 averaging time, $\Delta\Theta$: Predefined time interval for which the air quality characteristic is made representative.

NOTE 2 Every measured value obtained is representative of a defined interval of time, τ, the value of which always lies above a certain minimum due to the intrinsic properties of the measuring procedure applied. In order to attain mutual comparability of data pertaining to comparable objects, a normalization to a common, predefined interval of time is necessary. By convention, this normalization is achieved by transformation by means of a simple, linear and unweighted averaging process.

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Averaging of a series of discrete samples:

$$\hat{c}(\Theta|\Delta\Theta) = \frac{1}{K} \sum_{k=1}^{K} \hat{c}[\Theta_0 + (k-1)\tau|\tau] \qquad \dots (1)$$

where

$$\Theta_0 = \Theta - \Delta\Theta$$

$$K\tau = \Delta\Theta, \quad \tau \ll \Delta\Theta$$

Averaging of a continuous time series:

$$\hat{c}(\Theta|\Delta\Theta) = \frac{1}{\Delta\Theta} \int_{\Theta_0}^{\Theta} \mathrm{d}\Theta \; \hat{c}(\Theta|\tau) \qquad \dots (2)$$

In both cases, the original sample described by $\hat{c}(\tau)$ is linked to a representative interval of time τ, whereas $\hat{c}(\Delta\Theta)$, the result after application of the averaging process, is made representative for the interval of time $\Delta\Theta$ (just preceding Θ), the averaging time.

The averaging time, $\Delta\Theta$, is therefore the predefined and, by convention, common time interval for which the measured variable $\hat{c}$ is made representative in the sense that the square deviation of the original values, attributed to time intervals $\tau \ll \Delta\Theta$, from $\hat{c}$ over $\Delta\Theta$ is a minimum.

NOTE 3 The averaging process can alternatively be carried out by means of a special sampling technique (averaging by sampling).

3.2 continuously measuring system: System returning a continuous output signal upon continuous interaction with the air quality characteristic.

3.3 non-continuously measuring system: System returning a series of discrete output signals.

NOTE 4 The discretization of the output variable can be due to sampling in discrete portions or to inner function characteristics of the system components.

3.4 influence variable: Variable affecting the interrelationship between the (true) values of the air quality characteristic observed and the corresponding measured values, e.g. variable affecting the slope or the intercept of or the scatter around the calibration function.

3.5 reference conditions: Specified set of values (including tolerances) of influence variables delivering representative values of performance characteristics.

3.6 period of unattended operation: Maximum admissible interval of time for which the performance characteristics will remain within a predefined range without external servicing, e.g. refill, calibration, adjustment.

3.7 randomization: Drawing of numbers, from a population consisting of the natural numbers 1 to n, at random one by one successively without replacement until the population is exhausted, these numbers having been associated in advance with n distinct objects or n distinct operations which are then rearranged in the order in which the numbers are drawn.

The order of the objects or operations is then said to be randomized. (See ISO 3534-1.)

3.8 random variable: Variable which may take any of the values within a specified set of values and with which is associated a probability distribution. [ISO 3534-1]

3.9 variance function: Variance of the output variable as a function of the air quality characteristic observed.

3.10 warm-up time: Minimum waiting time for an instrument, after switching on, to meet predefined values of its performance characteristics stabilized in a non-operating condition.

NOTES

5 In practice, warm-up time can be determined by using the performance characteristic that is expected to need the longest time to stabilize.

6 In the case of manual procedures, the corresponding term is *run-up time*.

4 Symbols

a_0, a_1, a_2	Coefficients of the variance function model
b_0, b_1	Parameters of the estimate function for the calibration function
C	Air quality characteristic
c	Value of C
$\hat{c}$	Measured value at c
c_i	Value of C in the ith sample; this sample may be generated from reference material
c_0	Normalization factor for air quality characteristics; in this case $\lvert c_0 \rvert = 1$
Δc_i	Inaccuracy of C at c_i
$\bar{c}_\omega$	Weighted mean, with set of weights ω_k
$\mathrm{DEP}(\hat{c})_{IV_i}$	First-order dependence of the measured value on the ith influence variable at c
$\mathrm{DEP}(b_0)_{IV_i}$	First-order dependence of the intercept on the ith influence variable
$\mathrm{DEP}(b_1)_{IV_i}$	First-order dependence of the slope on the ith influence variable
$\mathrm{DEP}(x)_{IV_i}$	First-order dependence of the output signal on the ith influence variable
$D(b_0)$	Drift (see ISO 6879) of the intercept of the linear calibration function
$D(b_1)$	Drift of the slope of the linear calibration function
$D(\hat{c})$	Drift of the measured value, $\hat{c}$, at c
F	Statistic (cf. F-test)
F_x	x-quantile of the F-distribution
I_{IV_i}	Selectivity with respect to the ith influence variable
IV_i	ith influence variable
iv_i	Value of IV_i
Δiv_i	Difference of values of IV_i
L	Total number of measurements of instability test
LDL	Lower detection limit
M	Total number of samples generated by reference material within one calibration experiment
N_i	Number of values of the output variable at c_i
p_l, p_u	Estimate of the slope of the regression function of the output variable with time at $c = c_l$, $c = c_u$ respectively
RES_c	Resolution at $C = c$
R, r	Reproducibility and repeatability, respectively
$\hat{s}$	Estimate of the smoothed standard deviation of X at c
$\hat{s}^2$	Smoothed estimate of the variance of X (repeated measurements) at c
s_0	Normalization factor for the standard deviation; in this International Standard the value of s_0 is assumed to be 1
s_{b_0}, s_{b_1}	Estimate of the standard deviation of instability (see ISO 6879) of the intercept, and of the slope of the linear calibration function, respectively
s_c	Estimate of the standard deviation of instability at c
$s_{\hat{c}x}$	Estimate of the standard deviation of the experimentally determined calibration function (in units of the air quality characteristic)
$s_{\hat{x}c}$	Estimate of the standard deviation of the experimentally determined calibration function (in units of the output variable)
s_i	Estimate of the standard deviation of repeated x_{ij} at c_i; j repetition index
$\hat{s}_i$	Smoothed estimate of the standard deviation of repeated x_{ij} at c_i; j repetition index
s_r	Estimate of the repeatability standard deviation

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$t_{\nu;q}$	q-quantile of the t-distribution with ν degrees of freedom
TC	Test characteristic of Grubbs' outlier test
X	Output variable
x	Value of X
$\hat{x}$	Estimate of x
$\hat{x}_i$	Estimate of output signal at c_i
$\bar{x}_i$	Mean of the set of output signals at c_i
$x_{i,\text{extr}}$	Output signal at c_i with the highest absolute distance from $\bar{x}_i$
x_{ij}	jth output signal at c_i
$x_{\text{l};i}$, $x_{\text{u};i}$	Output signal after i time intervals at the lower and upper value of the air quality characteristic of the reference material
$\bar{x}_\omega$	Weighted mean of the whole set of output signals within the calibration experiment
β_0, β_1	Intercept and slope of the linear calibration function, respectively
Θ	Time
ν	Number of degrees of freedom in the calibration experiment
ν_1, ν_2	Number of degrees of freedom for the numerator of the F-distribution, respectively
$\omega = \omega(c)$	Continuous weighting factor gained by modelling s_i
ω_i	Weighting factor at c_i

5 Requirements

5.1 Description of the procedure

All steps of the measurement method such as sampling, analysis, postprocessing and calibration shall be described. Figure 1 illustrates schematically the steps to be followed in making a measurement or performing a series of calibration experiments in order to determine performance characteristics.

NOTE 7 Under certain conditions it may be suitable to test only one step or a selected group of steps of the measurement method. Under other conditions it may not be possible to include all steps of the measurement method. It is recommended to include as many steps as possible.

5.2 Specification of performance characteristics to be tested

The performance characteristics of the measurement method shall be specified in order of their relevance for the final assessment of accuracy. The descriptors of the calibration function, i.e. intercept, β_0, and slope, β_1, as well as their qualifying performance characteristics are vital. Those performance characteristics for which prior knowledge is available, and those pertaining influence variables covered by randomization, are of lesser importance and need not be determined.

5.3 Test conditions

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Perform the tests under explicitly stated conditions which must be representative of the operational measurements. When testing for statistical performance characteristics, all specified influence variables shall remain constant. When testing for performance characteristics describing functional dependencies, all influence variables shall remain constant except the one under consideration.

By convention, the statistical performance characteristics used in this International Standard are estimated throughout at the confidence level $1 - \alpha = 0{,}95$.

6 Test procedures

6.1 Averaging time (see 3.1)

The range of allowable averaging times is constrained by the requirement that the differences among subsequent output signals be mutually statistically independent. The corresponding minimum of the averaging time is determined by a specific performance (time) characteristic:

a) for continuously measuring systems: the response time;

b) for non-continuously measuring systems: the sample time (filling time, accumulation time etc.).

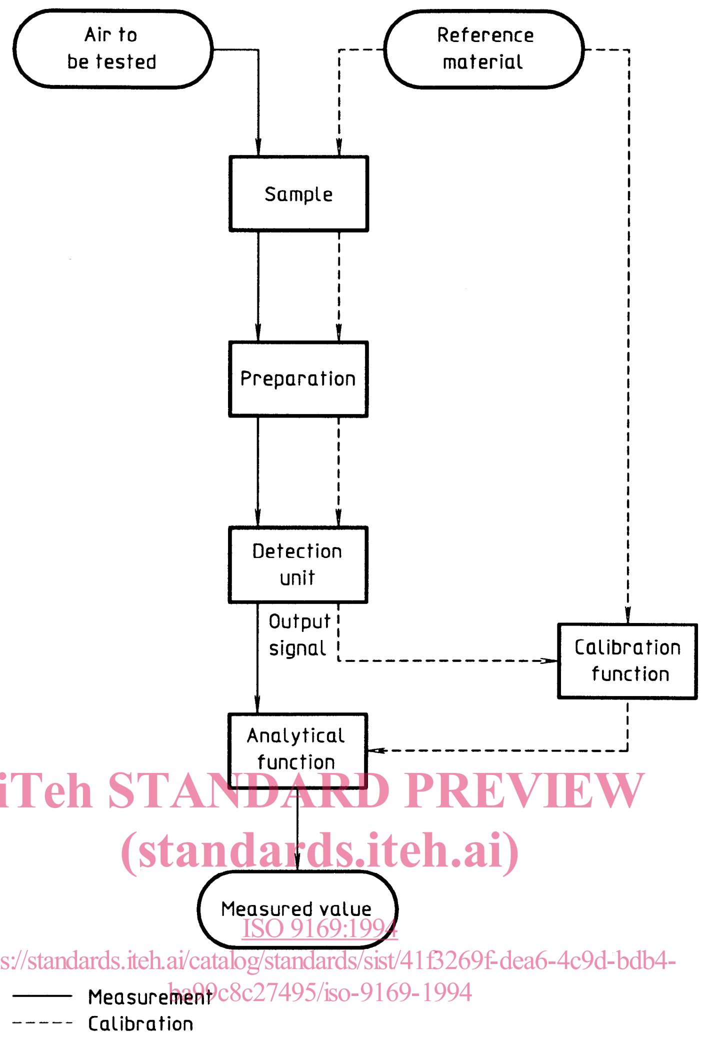

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Figure 1 — Scheme of the measurement and evaluation of performance characteristics

6.1.1 Continuously measuring systems

In order to establish response time, lag time, rise time and fall time, a step function of the air quality characteristic is input to the continuously measuring system. This may be done by abruptly changing the value of the air quality characteristic from e.g. 20 % to 80 % of the upper limit of measurement (see figure 2). These performance characteristics should be confirmed by an appropriate number of repetitions. If rise time and fall time differ, the longer one is to be taken for the computation of the response time. By convention, the minimum averaging time equals four times the response time.

6.1.2 Non-continuously measuring systems

The minimum averaging time is determined by the maximum of the sampling time, filling time or accumulation time, depending on the measurement method.

6.2 Functional and statistical performance characteristics[4)]

The performance characteristics to be determined are

a) those related to the calibration function and its stability under reference conditions;

b) those related to the dependence of the calibration function on influence variables.

4) The functional and statistical performance characteristics may be calculated on a computer using the TurboPascal program adjunct to ASTM Standard D 5280, available from ASTM, 1916 Race St., Philadelphia PA 19103-1187, USA.

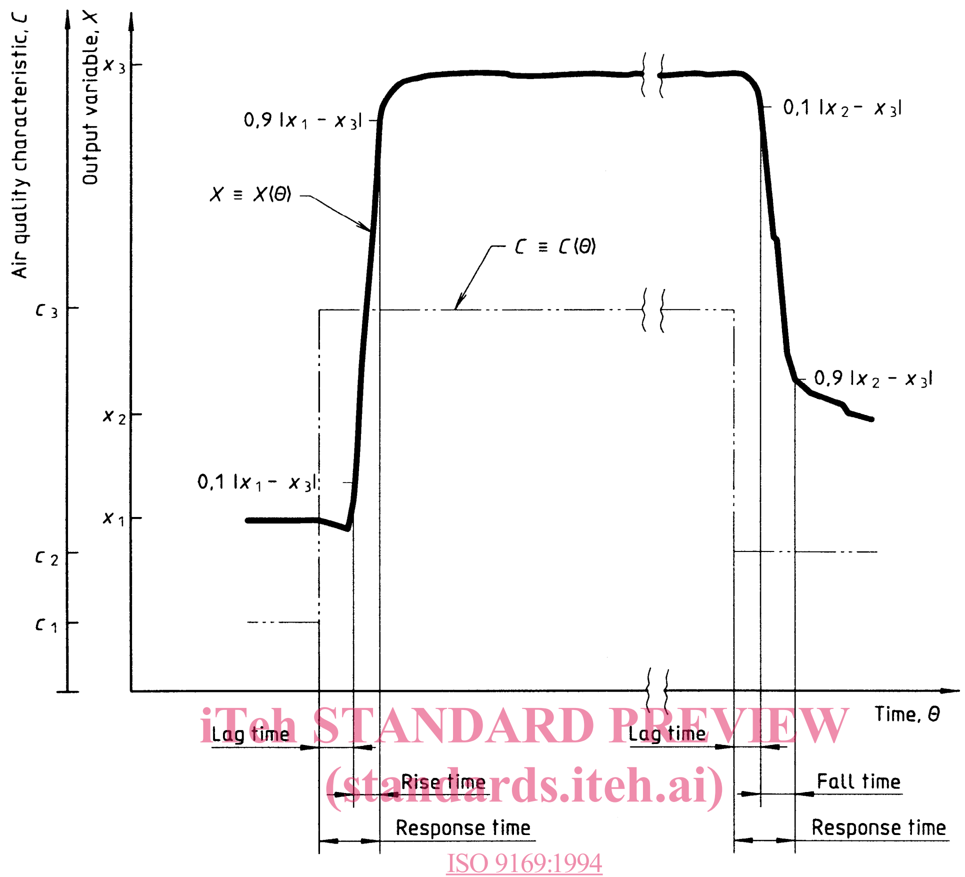

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Figure 2 — Diagram illustrating the performance (time) characteristics of a continuously measuring system

A linear calibration function is determined by its slope (sensitivity) and its intercept. Instability and the effects of influence variables are described by their impacts on the slope (sensitivity) and intercept.

All ouput signals evaluated throughout these tests shall be obtained after the measuring system has reached stabilized conditions.

6.2.1 Calibration

A calibration experiment for the evaluation of performance characteristics consists of at least ten repeated measurements at a minimum of five different values of the air quality characteristic.

If drift occurs, the duration of the calibration experiment shall be kept as short as possible. This may be accomplished by consecutive instrument readings at a certain value of the air quality characteristic and after a change in that value and stabilization, again consecutive instrument readings at that value, etc. (see figure 3). This is only valid in the absence of hysteresis or if hysteresis is negligible.

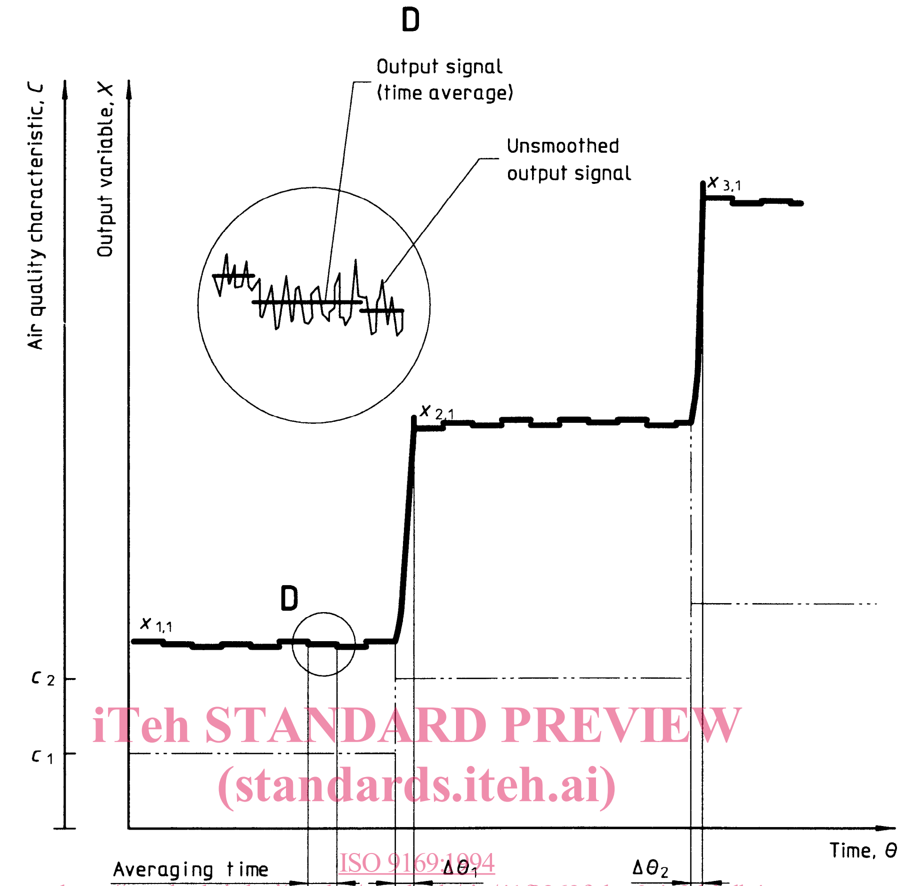
NOTE 8 Repetitions performed under reproducibility conditions (see ISO 5725) require a random sample of the population of the influence variables to be examined (randomization).

6.2.1.1 Elimination of outliers

Usually, experience helps to identify potential outliers. A less arbitrary way of detection of such potential outliers is given by combination of this experience with, e.g., Grubbs' test [1]. However, it should be clear that such a test identifies *potential* outliers. The underlying reasons may be statistical or due to system operation interferences. The latter presents sufficient grounds for elimination of the respective output signal (confirmation as an outlier).

Estimate the standard deviation s_i at c_i by:

$$s_i = \sqrt{\left[\sum_j x_{ij}^2 - \left(\sum_j x_{ij}\right)^2 / N_i\right] / (N_i - 1)} \qquad \ldots (3)$$



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x_{ij} jth time average over the interval of time $\Delta\Theta$ at the ith value of the air quality characteristic, generated by reference material

$\Delta\Theta_i$ Intervals of time during which unsmoothed output signals shall not be submitted to the averaging procedure and, thus, not be evaluated

Figure 3 — Example of a calibration experiment

At c_i, take the ouput signal $x_{i,\text{extr}}$ with the highest absolute distance from the mean output signal $\bar{x}_i$. Derive the test characteristic

$$\text{TC} = |x_{i,\text{extr}} - \bar{x}_i| / s_i \qquad \ldots (4)$$

where

$$\bar{x}_i = \left(\sum_j x_{ij} \right) / N_i \qquad \ldots (5)$$

and compare it with the tabulated value of Grubbs' two-sided outlier test (see annex A) to be taken as the critical value.

If TC exceeds the critical value, check if it is due to operational reasons, and if so, reject it. This procedure may be repeated; however, if more than 5 % of the number of output signals is rejected this way, this calibration experiment is not valid.

If operational reasons are not found for TC exceeding the critical value, the potential outlier may not be eliminated. In this case, verification of a basic test assumption or prerequisite is recommended.

6.2.1.2 Computation of the variance function

A central tool for the estimation of relevant performance characteristics is the variance function. Therefore, some guidelines for its computation, and the computation of related parameters are given.

Compute for each of the values c_i ($i = 1$ to M) of the air quality characteristic the variance s_i^2 of the output signals x_{ij} ($j = 1$ to N_i):

$$s_i^2 = \left[\sum_j x_{ij}^2 - \left(\sum_j x_{ij} \right)^2 / N_i \right] / (N_i - 1) \qquad \ldots (6)$$

Additionally, the dependence of s_i^2 on c is modelled [2] using

$$\log \frac{\hat{s}^2}{s_0^2} \approx a_0 + a_1 \sqrt{\frac{c}{c_0}} + a_2 \left(\sqrt{\frac{c}{c_0}} \right)^2 \qquad \ldots (7)$$

so the coefficients of this non-weighted second-order polynomial in $\sqrt{\frac{c}{c_0}}$ can be computed as:

$$a_2 = \frac{\left[Q_{(z^2,\, y)}\, Q_{(z,\, z)} - Q_{(z,\, y)}\, Q_{(z,\, z^2)} \right]}{\left[Q_{(z,\, z)}\, Q_{(z^2,\, z^2)} - \left(Q_{(z,\, z^2)} \right)^2 \right]}$$

$$a_1 = \frac{\left[Q_{(z,\, y)}\, Q_{(z^2,\, z^2)} - Q_{(z^2,\, y)}\, Q_{(z,\, z^2)} \right]}{\left[Q_{(z,\, z)}\, Q_{(z^2,\, z^2)} - \left(Q_{(z,\, z^2)} \right)^2 \right]} \qquad \ldots (8)$$

$$a_0 = \frac{\left(\sum_i y_i - a_1 \sum_i z_i - a_2 \sum_i z_i^2 \right)}{M}$$

with

$$Q_{(\zeta^m,\, \eta^n)} = \frac{\sum_i (\zeta_i^m\, \eta_i^n) - \left(\sum_i \zeta_i^m \right) \left(\sum_i \eta_i^n \right)}{M} \qquad \ldots (9)$$

Element $Q_{(\zeta^m,\, \eta^n)}$ is obtained by substituting ζ with z and η with z or y:

$$y_i = \log \frac{s_i^2}{s_0^2}\ ;\ z_i = \sqrt{\frac{c_i}{c_0}} \qquad \ldots (10)$$

An example of a variance function obtained this way is given in figure 4.

Consequently, the smoothed variance function $\hat{s}^2$ is obtained as:

$$\hat{s}^2 = \hat{s}^2(c) = s_0^2 \exp \left(a_0 + a_1 \sqrt{\frac{c}{c_0}} + a_2 \frac{c}{c_0} \right) \ldots (11)$$

The weighting factor, ω_i, at c_i ($i = 1$ to M), to be used later in the computation of the calibration function [1,2,3], is proportional to the inverse of the above variance,

$$\omega = \omega(c) = \frac{s_0^2}{\hat{s}^2} \qquad \ldots (12)$$

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6.2.1.3 Computation of the calibration function

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A linear calibration function [5]

$$x = \beta_0 + \beta_1\, c \qquad \ldots (13)$$

may be estimated by

$$\hat{x} = b_0 + b_1\, c \qquad \ldots (14)$$

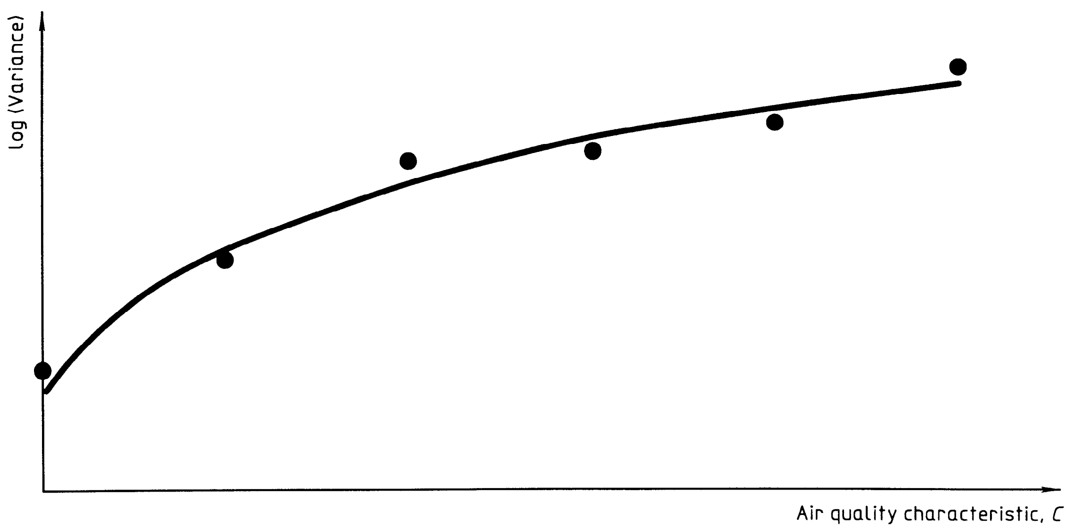

Figure 4 — Fit of the logarithm of the variance function

where

$$b_1 = \frac{\sum_i \sum_j \omega_i \, x_{ij} \, (c_i - \bar{c}_\omega)}{\sum_i N_i \, \omega_i \, (c_i - \bar{c}_\omega)^2}$$

$$b_0 = \bar{x}_\omega - b_1 \, \bar{c}_\omega \qquad \text{. . . (15)}$$

and

$$\bar{c}_\omega = \sum_i N_i \, \omega_i \, c_i \, / \sum_k N_k \, \omega_k$$

$$\bar{x}_\omega = \sum_i \sum_j \omega_i \, x_{ij} \, / \sum_k N_k \, \omega_k \qquad \text{. . . (16)}$$

In addition to the various standard deviations defined as descriptors for the mutual scattering of true values, measured values and output signals, there arises a special scatter to be attributed to the estimation process as a whole.

This scatter may be described by the following standard deviation [2]:

$$s_{\hat{x}c} = \sqrt{\frac{\sum_{i=1}^{M} \omega_i \sum_{k=1}^{N_i} (x_{ik} - \hat{x}_i)^2}{\left(\sum_{i=1}^{M} N_i\right) - 2}} \qquad \text{. . . (17)}$$

Sometimes the output signal is obtained after correction for the blank. When the blanks correspond to genuine zero samples, the corrected calibration function must pass through the origin. In this case the coefficient, b_1, reduces to:

$$b_{1;\mathrm{trf}} = \frac{\sum_i \sum_j \omega_i \, x_{ij} \, c_i}{\sum_k N_k \, \omega_k \, c_k^2} \qquad \text{. . . (18)}$$

The standard deviation, $s_{\hat{x}c}$, is invariant to the transformation, but the number of degrees of freedom changes to:

$$\nu_{\mathrm{trf}} = \left(\sum_{i=1}^{M} N_i\right) - 1 \qquad \text{. . . (19)}$$

6.2.1.4 Computation of the analytical function

Compute the analytical function by inverting the calibration function:

$$\hat{c} = \frac{x - b_0}{b_1} \qquad \text{. . . (20)}$$

6.2.1.5 Linearity

The hypothesis of linearity of the calibration function (see figure 5) is examined using the F-test [6]:

$$F = \frac{\left[\sum_i N_i \, \omega_i \, (\bar{x}_i - \hat{x}_i)^2\right] / \nu_1}{\left[\sum_i \sum_j \omega_i \, (x_{ij} - \bar{x}_i)^2\right] / \nu_2} \qquad \text{. . . (21)}$$

where

$$\nu_1 = M - 2$$

$$\nu_2 = \sum_i (N_i - 1)$$

If F does not exceed the tabulated value $F_{\nu_1, \nu_2; 1-\alpha}$ of the F-distribution for the one-sided test for the significance level $\alpha = 0{,}05$ (see annex B) to be taken as critical value, non-linearity is negligible, and the subsequent performance characteristics can be determined.

If F does exceed the critical value, the hypothesis of linearity must be rejected. The question whether non-linearity is substantial as compared to other uncertainties may be tested by determining whether the following inequality criterion holds:

$$\operatorname*{Max}_{i=1}^{M} \left\{ \frac{|\bar{x}_i - \hat{x}_i|}{2 \, s_i} \right\} < 1 \qquad \text{. . . (22)}$$

If the inequality criterion is met (see figure 5), the subsequent performance characteristics can be calculated. If the inequality criterion is not met, the determination of performance characteristics must be terminated. For the latter situation, the following procedure is recommended:

a) Examine the quality of reference material samples as a potential cause for non-linearity.

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