
**Air quality — Assessment of uncertainty of
a measurement method under field
conditions using a second method as
reference**

*Qualité de l'air — Évaluation de l'incertitude d'une méthode de mesurage
sur site en utilisant une seconde méthode comme référence*



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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

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.

International Standard ISO 13752 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 4, *General aspects*.

Annexes A, B and C of this International Standard are for information only.

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Introduction

Performance characteristics for air quality measuring methods are defined in ISO 6879. Corresponding test procedures are given in ISO 9169 except for accuracy, which is dealt with in this International Standard as measurement uncertainty following the concepts of the *Guide to the expression of uncertainty in measurement* [5].

The measurement uncertainty under field conditions is also covered in ISO 7935 and ISO 10849. However, the procedure given in these International Standards is limited to either the determination of a concentration-independent systematic deviation, assuming a concentration-independent dispersion, or a concentration-proportional systematic deviation, assuming a concentration-proportional dispersion.

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Air quality — Assessment of uncertainty of a measurement method under field conditions using a second method as reference

1 Scope

This International Standard specifies a method for assessing the measurement uncertainty of a calibrated measurement method (test method) applied under field conditions using a second method as a reference (reference method). The reference method may not necessarily be a legally prescribed measurement method. The measurement uncertainty is derived from measurements made in parallel on real samples by comparing the measured values of the test method with those of the reference method. The result is only valid within the range of the measurements obtained. The test is designed especially for method validation.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 6879:1995, *Air quality — Performance characteristics and related concepts for air quality measuring methods*.

ISO 9169:1994, *Air quality — Determination of performance characteristics of measurement methods*.

3 Symbols and abbreviated terms

For the purposes of this International Standard, the following symbols and abbreviated terms apply.

a_0, a_1, a_2	coefficients of the variance function
AQC	Air Quality Characteristic (usually concentration)
b_0, b_1	coefficients of the linear regression function or calibration function
F	F statistic
k	coverage factor
L	likelihood
ℓ	logarithmic likelihood
N, N_1, N_2	number of pairs (x_i, y_i) and number of pairs of subpopulation 1 and 2 respectively

$P(y_i)$	probability of y_i
r_i	residual at x_i
s, s_i	standard deviation as a function of AQC and at value x_i of AQC respectively
s'	transformed standard deviation as a function of AQC
$s_{a_0}, s_{a_1}, s_{a_2}$	standard deviation of a_0, a_1 and a_2 respectively
s_{b_0}, s_{b_1}	standard deviation of b_0 and b_1 respectively
s_x, s_y	standard deviation of all values x_i and y_i respectively
$s_{y_{\text{cor}}}$	standard deviation of the measured y -value after correction for the systematic error (bias)
$s_{\Delta y}$	standard deviation (uncertainty) of the systematic error (bias)
U	expanded uncertainty (coverage factor $k = 2$) as a measure of measurement uncertainty
X	variable of x -method
x, x_i	value of AQC and i -th value of AQC respectively
x'_i	transformed value x_i
$\bar{x}, \bar{x}_w, \bar{y}$	mean and weighted mean of all values x_i and mean of all values y_i respectively
Y	variable of y -method
y_i	measured value of y -method at x_i or output value of y -method at x_i
y'_i	transformed value y_i
$\hat{y}$	estimated value of Y at value x of AQC
$\hat{y}_i$	estimated value of Y at value x_i of AQC
y_{cor}	measured value of the y -method after correction for the systematic error (bias)
Δy	systematic error (bias) at value x of AQC
ε	random number from normal distribution with central value 0 and standard deviation 1
w_i	weighting factor at x_i

4 Principle

A number N of pairs of measured values $[(x_1, y_2), \dots, (x_N, y_N)]$ are obtained from parallel field measurements. The measured values from the reference method (x -method) are considered as true values. The difference between the values of a measurement pair is attributed to measurement deviation of the test method (y -method).

It is assumed that there is a linear relationship between the X and Y variable estimated by:

$$\hat{y} = b_0 + b_1 x \quad \dots(1)$$

The regression coefficients b_0 and b_1 can be calculated arithmetically if one of the following assumptions on dispersion of the y -values holds:

- standard deviation of the test method is independent of x (i.e. standard deviation is constant) and estimated by:

$$s^2 = a_0^2 \quad \text{or} \quad s = a_0 \quad \dots(2)$$

- standard deviation of the test method is proportional to x (i.e. coefficient of variation is constant) and estimated by:

$$s^2 = a_2^2 x^2 \quad \text{or} \quad s = a_2 x \quad \dots(3)$$

NOTES

1 The first assumption can be considered as fluctuations of the background or intercept value b_0 without fluctuations of the slope b_1 and the second as fluctuations of the slope without fluctuations of the background or intercept value.

2 The value of the coefficients of the regression function (estimation of bias) is not seriously affected by deviations from the assumption on the standard deviation. However, the estimated random part of measurement uncertainty heavily depends on the assumption.

The general variance function used in this International Standard accounts not only for the variability of intercept and slope but also for statistical noise, the standard deviation of which is proportional to the square root of the value itself (approximately proportional to the square root of x):

$$s^2 = a_0^2 + a_1^2 x + a_2^2 x^2 \quad \dots(4)$$

NOTES

3 Coefficients have been taken as squares because the coefficient rather than its square reflects the physical meaning.

4 The calculation procedure of the general variance function according to ISO 9169 cannot be used because repetitive measurements are not available.

The coefficients of this model [b_0 and b_1 of equation (1) and a_0 , a_1 and a_2 of equation (4)] cannot be calculated arithmetically. They are estimated iteratively on the criterion of maximum likelihood as an indicator of best fit (see figure 1). After selecting a set of start values for the coefficients and using the assumption on normality, the probability, $P(y_i)$, of every data point, (x_i, y_i) , belonging to the line can be calculated:

$$P(y_i) = \frac{1}{s_i \sqrt{2\pi}} e^{-\frac{(y_i - \hat{y}_i)^2}{2s_i^2}} \quad \dots(5)$$

The likelihood L is the mathematical product of the individual probabilities of y -values:

$$L = \prod_{i=1}^N \frac{1}{s_i \sqrt{2\pi}} e^{-\frac{(y_i - \hat{y}_i)^2}{2s_i^2}} \quad \dots(6)$$

The likelihood L is the indicator of fit. The coefficients are changed and the likelihood computed until a maximum value for L is obtained. The corresponding coefficients are the most likely coefficients for the regression model. For the determination by maximum likelihood a computerized optimization procedure is necessary.

The uncertainty of a measured value for any AQC value is derived from the regression function and the variance function respectively.

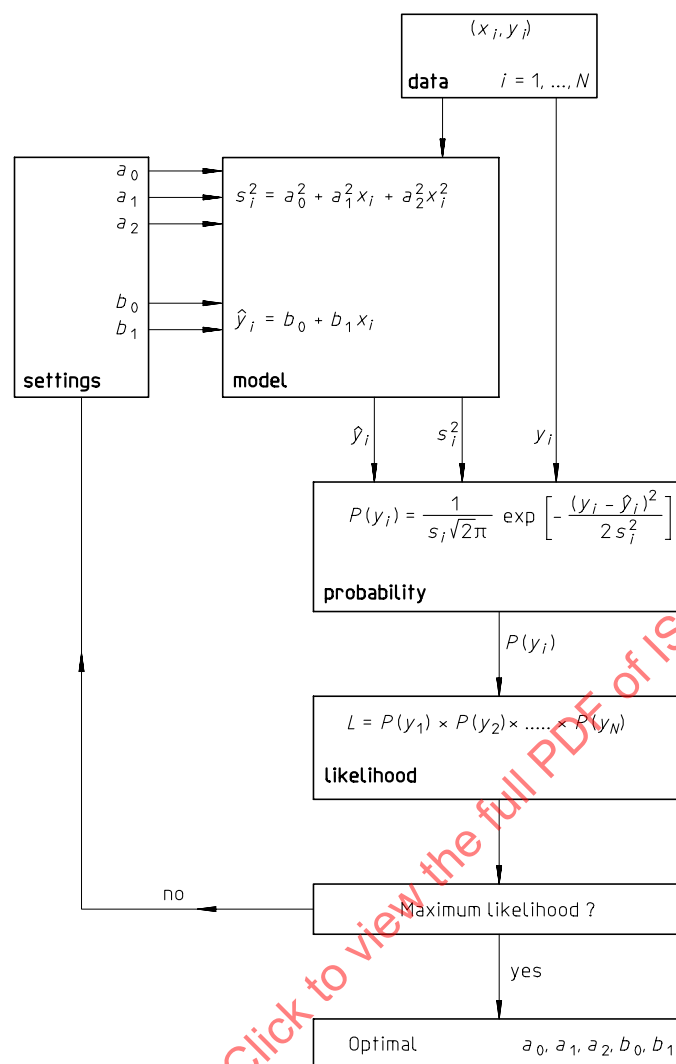


Figure 1 — Flow Scheme of maximum likelihood regression

5 Requirements

5.1 General

The method described in this International Standard requires that:

- a linear relationship exists between the variables compared; if the relationship is different but mathematically known the procedure may be adapted.
- the measurement errors of the test method are normally distributed.
- the measurement uncertainty of the reference method is insignificant compared to that of the test method; if not, it is falsely attributed to the test method and this will result in overestimating its measurement uncertainty.
- the impact of differences in composition between the air sampled by the two methods is negligible compared to the expected uncertainty of the test method; if not, this error component is falsely attributed to the test method and will result in overestimating measurement uncertainty.

The uncertainty of the coefficients calculated by this International Standard will be reduced by increasing the number of measurement pairs. Therefore, it is recommended that at least 30 measurement pairs be obtained if the general variance model described in this International Standard is applied.

5.2 Test method (y-method)

Specify all steps of the measurement method which will be subject to the assessment and execute the measurements as specified.

5.3 Reference method (x-method)

In view of the provisions and environmental conditions, e.g. interfering substances, temperature etc., to be expected at the test site, investigate whether the assumption is justified that the x-method yields insignificant uncertainty compared to the y-method. This investigation may be based on the properties of the measurement principle, literature data or results of laboratory tests or field tests.

Describe the x-method in detail and execute the measurements accordingly.

5.4 Test conditions

Make sure that the test conditions resemble the conditions under which the test method is going to be used (test period, range of the air quality characteristic, range of physical and chemical influence variables, and operational conditions). Describe the provisions and the environmental conditions at the test site.

The measurement equipment of both methods should be installed so that:

- the difference in composition between parallel samples is insignificant and,
- the equipment for one method does not influence that for the other.

5.5 Data processing

In the case of the general variance model, data processing requires computer facilities for finding the maximum fit (likelihood) by adjusting the values of a_0 , a_1 , a_2 , b_0 and b_1 .

6 Parallel measurements

Execute parallel measurements representative of the conditions under which the test method is going to be used. Record the pairs of measured values.

7 Graphical analysis of dispersion

The dispersion of measured values of a method is either constant or increases with the AQC value. An impression of the variance as a function of the AQC value can easily be obtained graphically by plotting, for all data pairs (x_i, y_i) , the absolute residuals $|r_i|$ against x_i where $r_i = y_i - \hat{y}_i$ and $\hat{y}_i$ is the predicted value for a conventional linear least-square fit:

- if the values of the residuals are independent of x_i go to 8.2;
- if the values of the residuals are proportional to x_i go to 8.3;
- if the values of the residuals are neither independent of nor proportional to x_i go to 8.4.

In those cases where the first or second relationships only applies to part of the range, the range must be curtailed accordingly. The coefficients of the regression function and variance function in 8.2 and 8.3 can be calculated arithmetically (simple variance model). Those of 8.4 require iterative computations which are only possible by means of a computer (general variance model).

8.2 and 8.3 provide procedures to test whether or not the assumption on the variance model is justified.

8 Estimation of coefficients of regression model

8.1 General

The assumed linear relationship between variables X and Y is estimated by linear regression:

$$\hat{y} = b_0 + b_1 x \quad \dots(7)$$

If dispersion about the regression line is independent of the AQC value, 8.2 applies:

$$s^2 = a_0^2 \quad \dots(8)$$

If dispersion about the regression line is proportional to the AQC value, 8.3 applies:

$$s^2 = a_2^2 x^2 \quad \dots(9)$$

Generally, if dispersion about the regression line is a monotonic function of the AQC value, 8.4 applies:

$$s^2 = a_0^2 + a_1^2 x + a_2^2 x^2 \quad \dots(10)$$

Adopt one of the variance models in this chapter.

8.2 Standard deviation constant

Compute the coefficient of the linear regression function:

$$b_1 = \frac{\sum_i x_i y_i - \frac{\sum_i x_i \sum_i y_i}{N}}{\sum_i x_i^2 - \frac{\left(\sum_i x_i\right)^2}{N}} \quad \dots(11)$$

$$b_0 = \bar{y} - b_1 \bar{x} \quad \dots(12)$$

Test whether or not the assumption of constant variance was justified:

- take N_1 measurement pairs from the upper end of the measurement range and N_2 pairs from the lower end with $N_1 = N_2 = N/3$;
- do not use the middle part of the range;
- compute the statistic F :

$$F = \frac{\left(\sum_{i=1}^{N_1} (y_i - \hat{y}_i)^2 \right) / (N_1 - 1)}{\left(\sum_{j=1}^{N_2} (y_j - \hat{y}_j)^2 \right) / (N_2 - 1)} \quad \dots(13)$$

- if F does not exceed the tabulated value $F_{N_1-1, N_2-1, 1-\alpha}$ of the F -distribution for the one-sided test for the significance level $\alpha = 0,05$ to be taken as the critical value, variance is assumed to be constant;
- if F exceeds the tabulated F value, proceed with 8.3 or 8.4.

Compute the variance function:

$$s^2 = a_0^2 = \frac{\sum_i (y_i - \hat{y}_i)^2}{N - 2} \quad \dots(14)$$

Compute the standard deviation of the coefficients b_0 and b_1 :

$$s_{b_0} = s_{b_1} \sqrt{\frac{\sum_i x_i^2}{N}} \quad \dots(15)$$

$$s_{b_1} = \frac{s}{\sqrt{\sum_i (x_i - \bar{x})^2}} \quad \dots(16)$$

8.3 Coefficient of variation constant

Transform the original data set (x_i, y_i) into a new data set (x'_i, y'_i) to obtain a linear relationship with constant variance by:

$$y'_i = \frac{y_i}{x_i} \quad \dots(17)$$

$$x'_i = \frac{1}{x_i} \quad \dots(18)$$

Compute the coefficient of the linear regression function:

$$b'_1 = \frac{\sum_i x'_i y'_i - \frac{\sum_i x'_i \sum_i y'_i}{N}}{\sum_i (x'_i)^2 - \frac{\left(\sum_i x'_i\right)^2}{N}} \quad \dots(19)$$

$$b'_0 = \bar{y}' - b'_1 \bar{x}' \quad \dots(20)$$

Test whether or not the assumption of constant variance in the transformed variables was justified:

- take N_1 measurement pairs from the upper end of the measurement range and N_2 pairs from the lower end with $N_1 = N_2 = N/3$;
- do not use the middle part of the range;
- compute the statistic F :

$$F = \frac{\left(\sum_{i=1}^{N_1} (y'_i - \hat{y}'_i)^2\right) / (N_1 - 1)}{\left(\sum_{j=1}^{N_2} (y'_j - \hat{y}'_j)^2\right) / (N_2 - 1)} \quad \dots(21)$$

- if F does not exceed the tabulated value $F_{N_1-1, N_2-1, 1-\alpha}$ of the F -distribution for the one-sided test for the significance level $\alpha = 0,05$ to be taken as the critical value, variance is assumed to be constant;
- if F exceeds the tabulated F value, proceed with 8.2 or 8.4.

Compute the variance function:

$$s'^2 = \frac{\sum_i (y'_i - \hat{y}'_i)^2}{N-2} \quad \dots(22)$$

Transform back the obtained values:

$$b_0 = b'_1 \quad \dots(23)$$

$$b_1 = b'_0 \quad \dots(24)$$

$$s^2 = a_0^2 x^2 = s'^2 x^2 \quad \dots(25)$$

Compute the standard deviation of the coefficients b_0 and b_1 :

$$s_{b_0} = \frac{\sum_i x'_i}{\sum_i (x'_i)^2} \sqrt{\frac{s'^2}{N} + s_{b_1}^2} \quad \dots(26)$$

$$s_{b_1} = s' \sqrt{\frac{\sum_i (x'_i)^2}{N \sum_i (x'_i - \bar{x}')^2}} \quad \dots(27)$$

8.4 General variance model

Select start values for the model coefficients b_0 and b_1 [equation (7)] and a_0 , a_1 and a_2 [equation (10)] where $a_0 \neq 0$.

Calculate for every measurement pair (x_i, y_i) :

$$\hat{y}_i = b_0 + b_1 x_i \quad \dots(28)$$

$$s_i^2 = a_0^2 + a_1^2 x_i + a_2^2 x_i^2 \quad \dots(29)$$

Calculate the logarithmic likelihood ℓ :

$$\ell = \sum_{i=1}^N \left(-\ln(s_i) - \frac{1}{2} \ln(2\pi) - \frac{(y_i - \hat{y}_i)^2}{2s_i^2} \right) \quad \dots(30)$$

Find the maximum value of ℓ iteratively by changing b_0 , b_1 , a_0 , a_1 and a_2 using an optimization procedure, e.g. the gradient method. As the coefficients a_0 , a_1 and a_2 appear in the equations quadratically, positive and negative values will do. Retain the positive value:

$$a_0 = |a_0| \quad \dots(31a)$$

$$a_1 = |a_1| \quad \dots(31b)$$

$$a_2 = |a_2| \quad \dots(31c)$$

Retain the values of b_0 , b_1 , a_0 , a_1 and a_2 corresponding to maximum L .

Compute the standard deviation of the coefficients b_0 and b_1 :

$$s_{b_0} = \sqrt{\frac{\sum_i \omega_i x_i^2}{\sum_j \omega_j \sum_i \omega_i (x_i - \bar{x}_\omega)^2}} \quad \dots(32)$$

$$s_{b_1} = \frac{1}{\sqrt{\sum_i \omega_i (x_i - \bar{x}_\omega)^2}} \quad \dots(33)$$

where:

$$\omega_i = \frac{1}{s_i^2} \quad \dots(34)$$

$$\bar{x}_\omega = \frac{\sum_i \omega_i x_i}{\sum_i \omega_i} \quad \dots(35)$$

The calculation procedure is illustrated by a spreadsheet program in Annex A and a corresponding example in Annex B.

NOTES

- 1 The terms $a_1^2 x$ and/or $a_2^2 x^2$ in the variance function may not be significant. This may be tested by fixing the assumed non-significant coefficient(s), a_1 and/or a_2 , to zero and repeating the procedure to establish the corresponding maximum ℓ . If the absolute difference between both ℓ values is less than 2 there is no significant difference between the models. Retain the simpler model.
- 2 Usually, a variance function of the form $s^2 = a_0^2 + a_2^2 x^2$ will do ($a_1 = 0$). This function reflects the tendency of dispersion to be constant at the lower end and to be proportional to the AQC value at the higher end of the measurement range. Only at a high number of measurement pairs might it be worthwhile to introduce three terms in the variance function.
- 3 If the relationship $y = f(x)$ is other than linear and its mathematical form known, equation (28) may be replaced by this mathematical function and its coefficients determined similarly by maximum likelihood regression.

9 Estimation of measurement uncertainty

The coefficients b_0 and b_1 are significantly different from the ideal values 0 and 1 respectively if:

$$|b_0| - 2s_{b_0} > 0 \quad \dots(36)$$

and

$$|b_1 - 1| - 2s_{b_1} > 0 \quad \dots(37)$$

If equations (36) or (37) show significance of corrections, then the systematic error (bias) at $X = x$ may be calculated within the range of measurements from:

$$\Delta y = b_0 + (b_1 - 1) x \quad \dots(38)$$

According to the procedures of the *Guide to the expression of uncertainty in measurement* [5], a systematic error arising from a recognized effect can be compensated for by applying a correction. A correction may only be applied if the values of influence variable(s) responsible for this systematic error are representative of the application. However, the uncertainty of the correction, which equals the uncertainty of the systematic error remains:

$$s_{\Delta y}^2 = s_{b_0}^2 - s_{b_1}^2 (x^2 - 2x\bar{x}_\omega) \quad \dots(39)$$

where:

a) Variance model 8.2:

$$\bar{x}_\omega = \bar{x} \quad \dots(40)$$

b) Variance model 8.3:

$$\bar{x}_\omega = \frac{\sum_i \frac{1}{x_i}}{\sum_i \frac{1}{x_i^2}} \quad \dots(41)$$

c) Variance model 8.4:

see equations (34) and (35).

Compute for $X = x$ the standard deviation of field measurements from the variance function:

$$s^2 = a_0^2 + a_1^2 x + a_2^2 x^2 \quad \dots(42)$$

Assuming a correction can be made for the systematic error Δy , the standard deviation of the corrected value y_{cor} is calculated according to:

$$\text{var}(y_{\text{cor}}) = \text{var}(y) + \text{var}(\Delta y) \quad \dots(43a)$$

$$s_{y_{\text{cor}}}^2 = s^2 + s_{\Delta y}^2 \quad \dots(43b)$$

The uncertainty of a single measurement at $X = x$ with a coverage factor $k = 2$ is given by:

$$U = 2\sqrt{s^2 + s_{\Delta y}^2} \quad \dots(44)$$

The coverage factor $k = 2$ corresponds to the Student factor for a 95% confidence interval and a normal distribution.

The *Guide to the expression of uncertainty in measurement* [5] recommends always correcting for systematic errors. Usually, instructions to determine and to correct for systematic errors are not given in the protocol. If corrections for systematic errors are not part of the y -method, these errors must be included in the measurement uncertainty. Applying the Guide's principle of expressing errors in terms of variances, the measurement uncertainty is given by:

$$U = 2\sqrt{s^2 + (\Delta y)^2} \quad \dots(45)$$

The numerical values for measurement uncertainty should always be accompanied with a statement on environmental and operational conditions under which the values were obtained.

Annex A (informative)

Template of spreadsheet to calculate regression and variance function

This annex shows an example of the calculation of the regression and variance function with a spreadsheet program based on Microsoft Excel® Version 5.0¹⁾. Insert the formulae of table A.1 in the corresponding cells of the blank spreadsheet form of table A.2.

Table A.1 — Formulae of the blank spreadsheet form

Cell	Contents
E5	=C5
E6	=C6
E7	=C7
E8	=C8
E9	=C9
E12	=SUM(G19:INDIRECT(ADDRESS(J3+18,7)))
F5	=SQRT((J5+J4^2)/(J5*J7))
F6	=SQRT(1/(J5*J7))
J3	=COUNT(B19:B2018)
J4	=SUMPRODUCT((B19:INDIRECT(J8))/(F19:INDIRECT(J10)))/J7
J5	=SUMPRODUCT(((B19:INDIRECT(J8))-J4)^2/(F19:INDIRECT(J10)))/J7
J6	=SUMPRODUCT((E19:INDIRECT(J9))^2/(F19:INDIRECT(J10)))/J7
J7	=SUMPRODUCT(1/(F19:INDIRECT(J10)))
J8	=ADDRESS(J3+18,2)
J9	=ADDRESS(J3+18,5)
J10	=ADDRESS(J3+18,6)
D19	=IF(ISNUMBER(C19),\$E\$5+\$E\$6*B19,"-")
E19	=IF(ISNUMBER(C19),C19-D19,"-")
F19	=IF(ISNUMBER(C19),\$E\$7^2+\$E\$8^2*B19+\$E\$9^2*B19^2,"-")
G19	=IF(ISNUMBER(C19),-(E19^2/F19)/2-LN(2.5066*SQRT(F19)),"-")

1) Excel® is the trade name of a product supplied by Microsoft. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Table A.2 — Text blank spreadsheet form

A	B	C	D	E	F	G	H	I	J	K
1	Version 1.2									
2	INPUT									
3	MODEL RESULTS									
4	Auxiliary variables									
5	Number of measurements									
6	Weighted average of x									
7	Weighted variance of x									
8	Weighted variance residuals									
9	Sum of weights									
10	Reference address									
11	Regression model									
12	$\hat{y} = b_0 + b_1 x$									
13	$s^2 = a_0^2 + a_1^2 x + a_2^2 x^2$									
14										
15										
16	DATA PAIRS									
17	Ref. method	Test method	Model y	Residue	Model var	In(prob)				
18	x	y	$\hat{y}$	$y - \hat{y}$	s^2					
19	1									
20	2									
21	3									
22	4									
23	5									
24	6									
25	7									
26	8									
27	9									
28	10									
29	11									
30	12									
16	INSTRUCTIONS									
17	1) Make sure that Add-In <Solver> is installed									
18	2) Always use a blank spreadsheet form (\$E\$5 to \$E\$9 will be overwritten)									
19	3) Insert N data pairs (xi,yi) in cells B19,C19 to B(N+18),C(N+18)									
20	4) Copy formulae D19 to G19 downwards to D(N+18) to G(N+18)									
21	5) Check / modify start values. If N > 2000 adjust J3									
22	6) Start tools item <Solver>. Wait for window <Solver Parameters>									
23	7) Default Solver Parameter settings are:									
24	* Set target cell: \$E\$12									
25	* Equal to: Max									
26	* By changing cells: \$E\$5:\$E\$9									
27	* Subject to the constraints: \$E\$7 >= 0.0000001									
28	Optional constraints are: \$E\$8 = 0 and/or \$E\$9 = 0									
29	(model simplifications)									
30	8) Resume solver operation: <Solve>									
	9) If model values for a1 or a2 are negative, retain the absolute value									