

International Standard



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Evaluation of the performance characteristics of gas analysers

Évaluation des caractéristiques des analyseurs de gaz

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Foreword

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Evaluation of the performance characteristics of gas analysers

1 Scope and field of application

This International Standard defines the main instrumental characteristics to be taken into consideration during the evaluation of gas analysers (continuous and discontinuous) and indicates the general principles and procedures of the test methods to be used in this evaluation. However, for some commercial transactions between manufacturers and users, the routine tests detailed in IEC document IEC/SC 66 D (Secretary) 20, *Expression of performance of gas analysers — Part 1: General* can be applied.

It can act as a guide to the official certification of gas analysers which comes within the scope of national legal metrology services.

2 General test conditions

2.1 Principle of an evaluation

Tests intended to determine the metrological characteristics consist in observing the operation and the indications of the instrument when it is receiving sequences of samples of gas of either known or stable and reproducible composition during the measuring period.

The environmental conditions under which the instrument is evaluated shall be known and recorded. Tests are applied to an instrument assembly that is defined and considered as a single entity (black box) for which the relation between the input and output is studied.

Generally separate aspects of the input/output relation of the instrument are studied by initiating an appropriate change in the input sample composition and observing the corresponding output signal. The procedure used should be suitably described and should be the most reliable possible. It should be based on the use of calibration gas mixtures, where these exist, the

preparation of which has been laid down by ISO/TC 158/SC 1, *Methods of preparation and definition of gas mixtures for calibration* (see the annex for a list of available documents on this subject).

A single evaluation is defined by the following items:

- description of the measuring assembly;
- installation of the analyser;
- definition of measuring sequences;
- environmental conditions;
- test mixtures;
- calibration procedure,

and depends **strongly** in particular on the definition of the **measuring sequence** within time.

These conditions are fixed at the beginning of the evaluation procedure not too far from usual operating conditions.

The principle of processing of the obtained data is given for every characteristic, without fixing in detail the acquisition and processing methods.

The values obtained are influenced by the imperfections of the test methods, including acquisition and processing of the data, and only represent estimates of those characteristics within the capabilities of the test equipment.

The significance of the results of tests carried out on a single analyser during a short period of time is obviously limited. It is indeed possible that for a given apparatus the results vary as a function of time and that these results are not always identical for two sets of apparatus of the same type. If it can be done, a repetition of tests enables this drawback to be limited.

2.2 Description of the measuring assembly to be evaluated

Gas analysers are generally complex measuring trains consisting of

- a system for sampling and transfer lines;
- a sensing cell or transducer;
- a system for processing the signals (for example an amplifier);
- an output assembly for reading, recording or data processing.

The same type of analyser can be used in many different applications with appropriate modifications of the measuring assembly. For a given evaluation all the separate points of the measuring assembly listed above shall therefore be clearly stated.

2.3 Installation of the analyser

Before operating the analyser, the user shall comply with the manufacturer's operating instructions particularly with regard to the installation of equipment and the quality and quantity of the consumable products necessary. Evaluation of the analyser shall furthermore be performed taking account of the warm-up time.

2.4 Continuous and discontinuous analysers — Definition of measuring sequences

According to the characteristic to be evaluated, it may be necessary to distinguish between analysers with continuous and discontinuous response.

For analysers with a continuous response, each measurement from 4.2 onwards shall be carried out under stable conditions, i.e. after a waiting time t_1 which is long enough for the analyser to become stable. The value of measurement is taken as the mean value of the signal over a time interval t_2 ; t_1 and t_2 are fixed and recorded by the experimenter, they are chosen taking account of the dynamic response characteristics in order to obtain a significant measurement under stationary conditions.

The mean of the signal obtained over the time interval t_2 defines, by convention, an independent measurement. Each independent measurement is obtained after performing a complete cycle determined on the basis of t_1 and t_2 .

For analysers with a discontinuous response, each measurement will be carried out during an interval of time corresponding to a specified number of sequences and located beyond the response time.

2.5 Environmental conditions

According to the characteristic to be evaluated, tests shall be carried out either under laboratory conditions, i.e. environmental conditions considered as stable and with little constraint, or under extreme environmental conditions simulated in the laboratory. In the two cases, the conditions are known and reported, and if possible fixed. Tests in the field are not covered by this International Standard.

2.5.1 Tests under laboratory conditions

The reference conditions for testing shall be within the following limits:

- temperature: between 17 and 29 °C;
- relative humidity: between 45 % and 75 %;
- pressure: local atmospheric pressure;
- electrical supply.

The conditions of supply (system voltage, supply frequency, distortion of the supply with alternating current) can be selected from one of the categories of use given below (in accordance with IEC Publication 359, *Expression of the functional performance of electronic measuring equipment*).

However, under certain special conditions (apparatus operating conditions, national regulations) different values may be chosen.

2.5.1.1 System voltage (with distortion of the wave-form)

		Direct or alternating current (effective value)	Alternating current (peak value)
Reference value		Nominal voltage	Nominal voltage
Tolerance on reference value		± 1 %	± 2 %
Nominal region of operation	I	± 10 %	± 12 %
	II	– 12 % to + 10 %	– 17 % to + 15 %
	III	– 20 % to + 15 %	– 30 % to + 25 %
Limit area of operation		Identical with the nominal region of operation, unless otherwise specified.	

2.5.1.2 Supply frequency

Reference value: nominal frequency

Tolerance on reference value: $\pm 1\%$

Nominal region of operation I and II:
nominal value $\pm 5\%$

Nominal region of operation III:
nominal value $\pm 10\%$

For each measurement, the value of each of these parameters, together with their variations (as low as possible), shall be recorded and shall be compatible with the sensitivity of the analyser for assessment with regard to these parameters.

The characteristics evaluated under these conditions include the characteristics defined in 4.1 to 4.8.

2.5.2 Operating tests under extreme environmental conditions simulated in the laboratory

These tests may relate to the following effects:

- a) apparatus parameters:
 - atmospheric pressure,
 - temperature,
 - relative humidity,
 - supply voltage and frequency, and supply interruptions,
 - vibrations,
 - shocks,
 - electrostatic discharges, electromagnetic interference,
 - etc.
- b) sample parameters:
 - flow, pressure, temperature, moisture content, etc.

The choice of parameters and of their extreme values depends upon the apparatus concerned and its future conditions of use.

2.6 Test mixtures

According to the characteristics to be evaluated, the concentrations of test mixtures shall be either known with their uncer-

tainty range or stable within a range which is approximately known. For the preparation of these test mixtures, references shall be made to the documents listed in the annex.

3 Gauging¹⁾

Prior to any evaluation, the analyser shall be gauged under reference conditions with calibration gas mixtures prepared according to the methods standardized by ISO/TC 158/SC 1; the positions of different controls (measurement range, potentiometers, flow, etc.) will be detailed and as far as possible shall not vary from the beginning to the end of the evaluation (if this is not the case, any change shall be noted).

4 Characteristics to be evaluated

4.1 Response time — lag time — rise time (or fall time)

4.1.1 Definitions

4.1.1.1 response time: Time interval from the instant at which a step change of sample concentration occurs at the input of the analyser to the instant at which the output reading reaches a level corresponding to 90 % of the final change in output reading.

4.1.1.2 lag time: Time interval from the instant at which a step change of sample concentration occurs at the input of the analyser to the instant at which the output reading reaches a level corresponding to 10 % of the final change output reading.

4.1.1.3 rise time (or fall time): Difference between the response time and the lag time.

4.1.2 Principle of the test method

A concentration C_1 is sent to the analyser, its response is Y_1 , then the input concentration is instantaneously changed from C_1 to C_2 : the response of the analyser then changes from Y_1 to Y_2 .

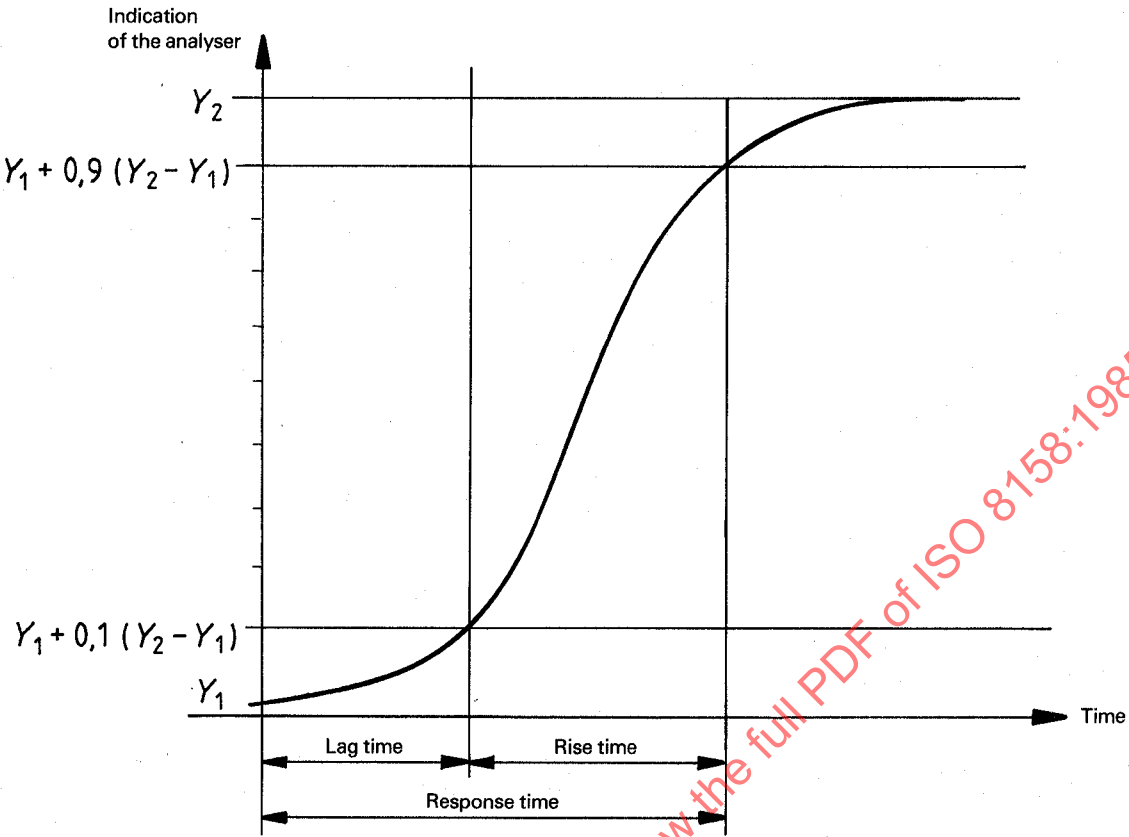
The response time is the time interval from the instant at which the change $C_1 \rightarrow C_2$ occurs to the instant at which the output reading reaches $Y_1 + 0,9 (Y_2 - Y_1)$.

1) This term is used according to the definition given in: BIPM, IEC, ISO, OIML. *International Vocabulary of Basic and General Terms in Metrology*. Geneva, ISO (1984):

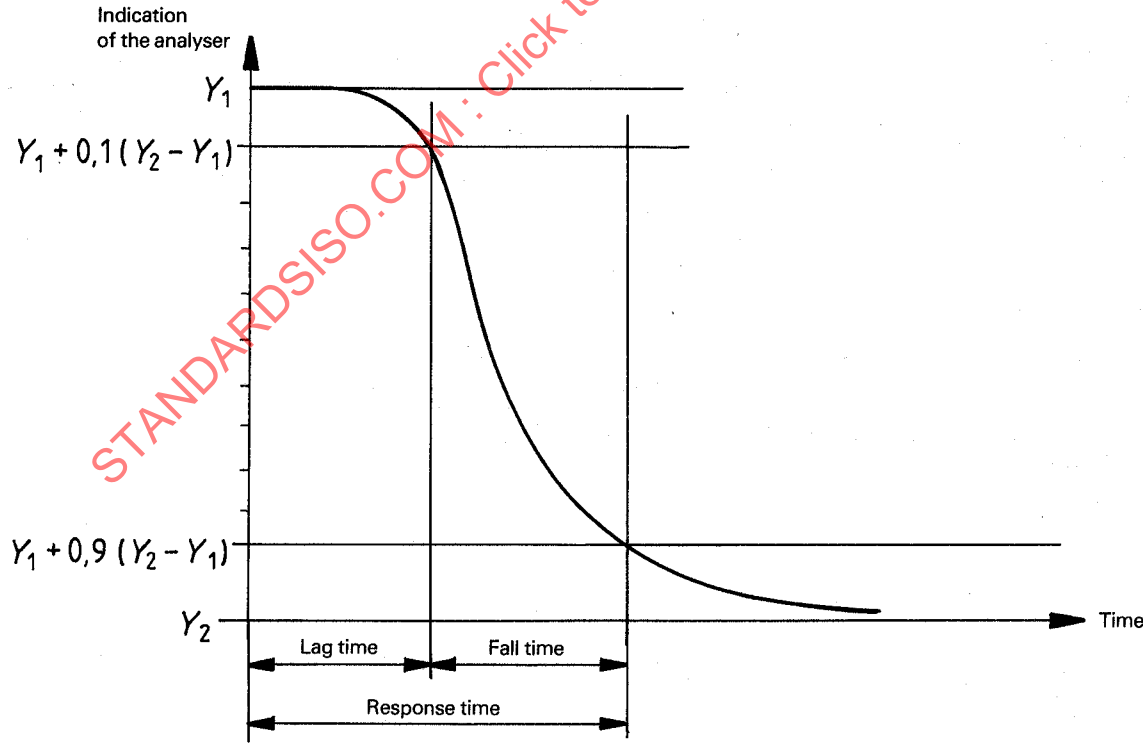
gauging (of a measuring instrument)

The operation of fixing the position of the gauge marks or scale marks of a measuring instrument (in some cases of certain principal marks only), in relation to the corresponding values of the measurand.

NOTE — "Gauging" should not be confused with "calibration".



a) Increasing step change in input



b) Decreasing step change in input

Figure 1 — Diagram illustrating response time, lag time and rise time

The lag time is the time interval from the instant at which the change $C_1 \rightarrow C_2$ occurs to the instant at which the output reading reaches $Y_1 + 0,1 (Y_2 - Y_1)$.

The uncertainty of these measurements depends on the stability of the response at the concentrations chosen. Therefore, and in order to calculate the response time better, if necessary, the operator is advised to carry out several determinations in the same operating conditions; the concentrations between which the response time and the lag time are determined shall be noted.

In addition, the operator will carry out other measurements with a small jump in concentration and with as large a jump in concentration as possible within the concentration range which can be measured, and he will also use increasing and decreasing concentrations when it corresponds to the normal working conditions of the analyser.

The aim of these experiments is to find out more about the variations in the response time of the analyser according to the operating conditions. The response time shall always be determined whatever the nature of the response of the analyser (continuous or discontinuous response); lag time and rise time are, on the contrary, generally less important characteristics than the response time and, in the case of discontinuous response type analysers, they may lose their significance. In the case of an appliance with discontinuous response, the response time will depend on the moment at which the instantaneous step concentration occurs at the input to the analyser.

4.1.3 Summary of information to be given

When the response time is used in a document describing the performance characteristics of an analyser or a measuring assembly, at least the following information shall be given:

- sample flow rate;
- values of the initial and final concentrations;
- stability of these concentrations;
- reading frequency of the analyser output signal during the measurement;
- method used to estimate the final value of the analyser output signal;
- number of measurements carried out.

4.2 Drift

4.2.1 Definition

drift: Change of the indications of an analyser, for a given level of concentration over a stated period of time, under reference conditions which remain constant.

It is necessary to distinguish the zero drift which concerns the operation of the instrument with samples of zero or low concentration from the drift considered at one or several levels of concentration.

4.2.2 Principle of the test method

One or more gas mixtures with constant or reproducible concentrations and one gas with zero or low concentration are used. These various gas mixtures, therefore different concentrations, are introduced into the analyser input. The sequence is reproduced periodically (see figure 2). Linear regressions, as a function of time, are made respectively for the indications corresponding to each level of concentration. The slopes of the linear regressions provide an estimation of the drift at each level.

The linear regression is given by the following equation:

$$Y = A + Bt$$

where

Y is the indication (not corrected by the indication obtained with the zero gas) obtained with time t ;

$$A = \frac{\sum Y - B \sum t}{n}$$

$$B = \frac{n \sum t Y - (\sum t) (\sum Y)}{n \sum t^2 - (\sum t)^2}$$

n is the number of measurements.

There are two different approaches to determine whether the drift is significant

- a) By use of the coefficient of linear correlation, r :

$$r = \frac{n \sum t Y - (\sum t) (\sum Y)}{\sqrt{[n \sum t^2 - (\sum t)^2] [n \sum Y^2 - (\sum Y)^2]}}$$

For n couples (response, time) and for a probability of 95 %, the correlation and therefore the slope are only significant if r is greater than $r_{95\%}$ given by the tables. The drift is therefore estimated by B .

- b) By use of the confidence interval

$$s = \sqrt{\frac{\sum (Y - At - B)^2}{n - 2}}$$

The drift is expressed by the calculated slope B of the regression line. Whether drift is significant is tested by the slope of the regression line being statistically different from zero.

The 95 % confidence interval of the slope b is given by

$$B - \frac{t_{0,975} \cdot s}{\sqrt{\sum t^2 - (\sum t)^2/n}} < b < B + \frac{t_{0,975} \cdot s}{\sqrt{\sum t^2 - (\sum t)^2/n}}$$

If the confidence interval of b does not include the value zero, drift is significant.

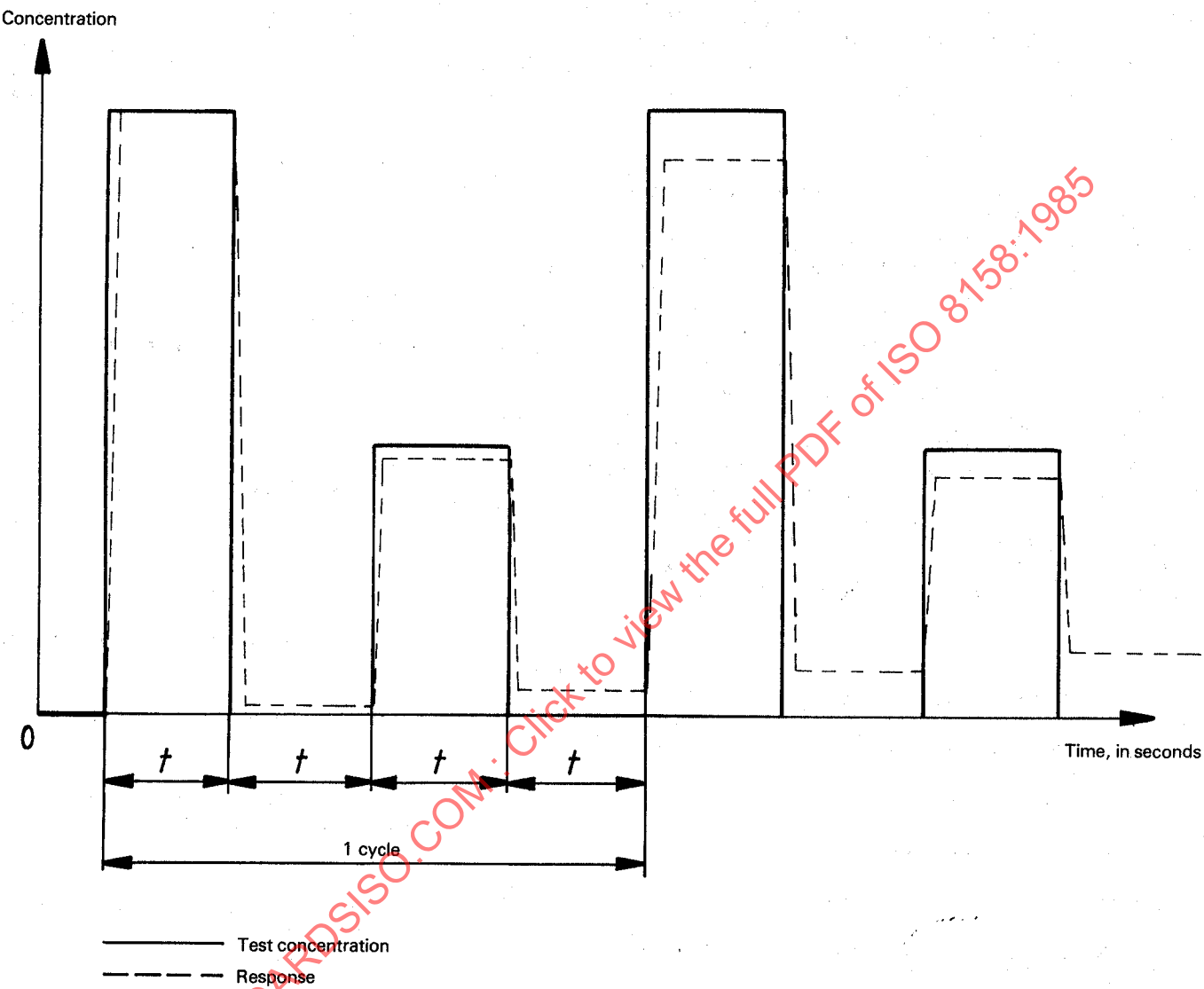


Figure 2 — Diagram illustrating drift

4.2.3 Precautions to be taken during the tests

4.2.3.1 The indication of the instrument shall be able to vary freely, in particular around zero, or otherwise it shall be shifted by a constant value which will then be subtracted from the reading to give operational indications.

4.2.3.2 The factors of influence and in particular the environmental conditions of the instrument are fixed and defined throughout the duration of the tests.

The conditions shall be in accordance with those given in 2.5.1 and can be achieved in a climatic chamber. If certain factors cannot be fixed

- either corrections will be able to be made to the indications of the instrument being tested, according to the values for these factors,
- or the value determined will also include the influence of these factors. This is generally the case for atmospheric pressure which can affect the sampling circuit and the measuring cell.

4.2.3.3 To avoid the possible influence of the discrimination error on the analyser response, a succession of different concentrations of the gas mixtures (see figure 2) is supplied to the input of the sampling line and therefore the concentration is not kept constant throughout the tests.

4.2.3.4 Each concentration considered shall be as constant as possible, in view of the accuracy required, during a period as long as the envisaged test requires.

In order to obtain the best possible stability of each of the gas mixtures used in succession, two methods will be used:

- where the gas mixtures have been prepared using a static method, the quantity of gas for each of the concentrations used shall be sufficient to feed the sampling circuit of the instrument throughout the test;
- where the gas mixtures are prepared using a dynamic method, as many circuits or generators, giving a continuous supply, can be used as there are concentrations. The flows are therefore alternately directed towards the analyser or towards a bypass circuit.

4.2.3.5 In order to save test gases, during the time intervals between the measuring time periods, another gas, whose concentration is approximately known and preferably within the working range, may be passed through the instrument.

4.2.3.6 As the drift is considered as a systematic evolution over a given period of time depending on the conditions of use of the instrument (period considered without any intervention or adjustment directly influencing the indications), it is necessary to ensure that the indications to be used for calculation do not include any slow periodic variations during the test period which could affect the linear regression to be made.

Those instabilities characterized by periodic variations, which are not part of the drift, may be due to:

- external causes; it is then a question of the effect of factors of influence, this will generally be the case of diurnal variations for example, which shall be taken into account during the test;
- causes internal to the instrument; generally a very rare case which, according to the period and the magnitude of the variations may lead to insignificant linear regression. In this case, it is necessary to perform a special study in order to characterize such a functioning which in itself is unsatisfactory due to the technology of the instrument.

4.2.4 Summary of information to be given

For each level of concentration, the following shall be given:

- duration of the test;
- number of points recorded, n ;
- slope of regression in relation to time, B ;
- ordinate at the origin, A ;
- coefficient of correlation in time, r .

COMMENT — When carrying out this test, it can also be of interest to record, for each level of concentration, the arithmetic mean of the instrument readings and the estimated relative standard deviation, s , of the instrument readings which can be used to characterize the fluctuations in the response during the test.

4.3 Calibration curve (repeatability of indications, intrinsic error)

Having fixed the parameters of adjustment of the analyser by the gauging operations, the errors on the indications are estimated with the aid of the test mixtures (calibration gas mixtures) considered with their ranges of uncertainty.

4.3.1 Terminology

The calibration curve is the graphic representation of the indications noted as a function of the concentrations to be measured.

From the dispersion of the indications obtained at a test concentration, the error of repeatability of the indication at this level of concentration is estimated.

The intrinsic error, taken as the possible significant difference between the indication and the test concentration, is estimated from a series of indications obtained at each test concentration.

At each level of test concentration, these two criteria — repeatability and intrinsic error — are representative of the calibration obtained after a given calibration operation. There

may be another cause of random error, connected with the repeatability of the calibration operations: this is estimated separately from the dispersion of the series of indications, each series being obtained, following a separate calibration operation, on the same test mixture.

4.3.2 Principle of the test methods

As the calibration curve is not available in its entirety, an approximation is determined with the aid of p test concentrations C , known with their uncertainties, the distribution of which over the measurement scale can be fixed as a function of the aims of the evaluation.

For each of these test concentrations, n measurements ($n > 10$) are carried out supplying n results.

The calibration curve is the representation in the diagram C, Y of the relation linking these two values: the response of an ideal instrument, without intrinsic error, is represented by the first bisectrix of the equation $Y = C$, ideal line of response.

The procedure to be followed takes into account the following points:

4.3.2.1 Operation following defined cycles of concentrations is recommended to provoke variations in response from the analyser and to avoid the possible effect of discrimination error.

4.3.2.2 The rectangle of error is determined at each level C of test concentration, taking into account

- the uncertainties ΔC on C , fixed by the preparation method or verification method of the calibration gas mixture;
- random dispersions ΔY of the response Y_C at level C .

It is assumed that the distribution of Y is normal with estimated standard deviation $s_{Y,C}$. Thus, the confidence interval P_1 , at 95 % probability, around the mean, $\bar{Y}_C$, is given by

$$\bar{Y}_C \pm s_{Y,C} \times \frac{t}{\sqrt{n}} = Y_C \pm \Delta Y$$

where t is the value of the Student-Fischer coefficient for $n-1$ degrees of freedom and a probability of 0,975. For $n = 10$

$$\frac{t}{\sqrt{n}} = 0,72$$

4.3.2.3 Thus, in the vicinity of each test concentration (see figure 3) with regard to the line of response I , within the errors due to the test methods used, are obtained

- a) An estimate of standard deviations σ_R of repeatability,¹⁾ the confidence interval of which at 95 % probability, for

$n < 30$, usually adopted, while assuming normal distributions in Y , is determined by

$$s_{Y,C} \sqrt{\frac{n-1}{\chi_{0,975}^2}} < \sigma_R < s_{Y,C} \sqrt{\frac{n-1}{\chi_{0,025}^2}}$$

where $\chi_{0,975}^2$ and $\chi_{0,025}^2$ are the values of χ^2 (Pearson's law) for $n-1$ degrees of freedom for probabilities of 0,975 and 0,025, respectively.

For $n = 10$

$$0,69 s_{Y,C} < \sigma_R < 1,83 s_{Y,C}$$

b) An estimate of the possible intrinsic error Δ_b obtained in the following way:

- 1) if the line of response ($Y = C$) intersects the rectangle of error, the uncertainties of the test method do not allow a significant intrinsic error to be detected;

- 2) if the line of response ($Y = C$) does not intersect the rectangle of error (as in figure 3) a significant intrinsic error is revealed. It is situated between two limits, calculated from the diagram, corresponding, respectively, to the point of the rectangle closest to and farthest away from the straight line I :

- in absolute value

$$\bar{A}a < \Delta_b < \bar{B}b$$

- in relative value

$$\frac{\bar{A}a}{Y} < \Delta_{br} < \frac{\bar{B}b}{Y}$$

COMMENT — The uncertainties of the test method, in particular $\Delta C/C$, have a decisive effect on these estimations in particular with regard to low concentrations where difficulty may be experienced in revealing significant intrinsic errors.

4.3.3 Summary of information to be given

For each concentration level, the following shall be given:

- number of measurements;
- mean value of the indications;
- estimated standard deviation;
- confidence interval of the standard deviation of repeatability;
- any intrinsic error.

1) For repeatability definition, see ISO 7504, *Gas analysis — Vocabulary*.

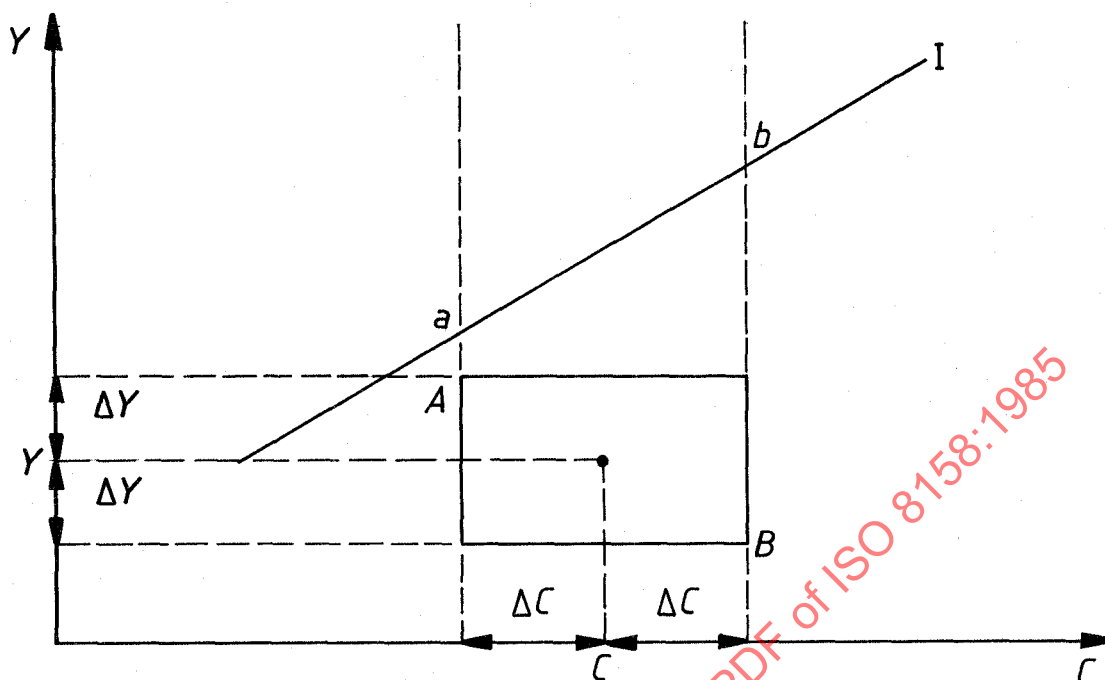


Figure 3 — Diagram illustrating the rectangle of error

4.4 Measurement threshold

4.4.1 Definition

measurement threshold, C_m : The smallest concentration which can effectively be measured. Quantitatively, it is defined as the concentration C_m for which a standard deviation of repeatability σ_R of 0,25 in relative value is obtained.

In the case of a large number of tests, the measurement threshold is the concentration for which 95 % of indications Y are higher than 95 % of the indications centred on the mean, obtained with a zero concentration. Assuming the distribution of the indications to be normal, with a standard deviation σ , gives

$$C_m = 4 \sigma \text{ in the case of a response without intrinsic error}^1)$$

or

$$C_m = \frac{4 \sigma}{1 + \Delta_{br}}, \Delta_{br} \text{ being the intrinsic error in relative value (see 4.3).}$$

At the level of concentration C_m , the relative error is approximately ± 50 % at the 95 % level of probability and for an independent measurement.

4.4.2 Principle of the test method

The confidence interval of σ is evaluated from the estimated standard deviation s , obtained on n consecutive indications Y , at a sufficiently low concentration C ($5 < C/s < 10$) so that the variations of σ between C and C_m with the uncertainty on σ given by its bilateral confidence interval at 95 % probability, can be disregarded.

$$s \sqrt{\frac{n-1}{\chi_{0,975}^2}} < \sigma < s \sqrt{\frac{n-1}{\chi_{0,025}^2}}$$

For $n = 10$

$$0,69 s < \sigma < 1,83 s$$

The evaluation of the measurement threshold involves, therefore, an evaluation of the dispersion of the indications at various concentrations, in order to find the conditions $5 < C/s < 10$. A convenient method is to take for the value of s the value found with the zero gas or the lowest concentration considered for the calibration curve.

The precautions to be taken are the same as those specified in 4.3 for the calibration curve.

1) In fact, $1,96 \sigma$ arising from the bilateral distribution of indications for $C = 0$ and $1,65 \sigma$ arising from the distribution for C_m (unilateral interval, 95 % area of probability blocked towards the increasing C). Their sum $3,61 \sigma$ is rounded up to 4σ .

4.4.3 Summary of information to be given

The following shall be given:

- estimation of the measurement threshold;
- and, for each concentration level,
- number of measurements;
- mean value of the indications;
- estimated standard deviation;
- confidence interval of the standard deviation of repeatability;
- any intrinsic error in relative value.

4.5 Memory effect

4.5.1 Definition

memory effect: A phenomenon which is revealed by a significant variation (momentary or otherwise) in the indications after the apparatus has been subjected to very high concentrations.

This phenomenon is characterized for the concentrations considered, on the one hand by the deviation of reading in a given time and, on the other hand, by the delay before a new indication from the instrument can be obtained.

COMMENT — The indication of the measuring instrument can be affected by previous samples. The characteristics "response time", "lag time", "rise time", only allow the dynamic characteristics of the instrument to be taken into account in conditions similar to the mean conditions of use of the instrument. The development of the concentration of the sample may involve extreme conditions (duration, level) liable to cause an acute appearance of hysteresis and, therefore, to impair at least temporarily the use of the measuring instrument.

4.5.2 Principle of the test method

Two sources of calibration gas mixtures of respective concentrations C_1 and C_2 can be connected alternatively to the calibration circuit of the instrument under study.

Concentration C_1 is low, the indication corresponding to the measuring sequence ($t_1 + t_2$) as defined in 2.4 is designated by Y_1 , obtained when the calibration curve is drawn.

Concentration C_2 is high, the level will be fixed according to the considered application for the instrument and shall not exceed any limit given by the manufacturer. The sampling circuit is then connected to mixture C_2 for the duration of the measuring sequence ($t_1 + t_2$).

Concentration C_1 is again introduced into the sampling circuit, in a rapid operation with a negligible transition time in the process under consideration.

After a delay time t_1 and while respecting the integration time t_2 Y_t is then recorded as a function of time.

The operation is repeated n times ($n > 6$).

From this is taken $\bar{Y}_{t_1}$, the mean of n tests, with its confidence interval at 95 % probability ($st/\sqrt{n}$), and from this is deduced:

- any significant deviation of the indication due to the corresponding hysteresis effect:

$$Y_{t_1} - Y_1$$

or relative deviation expressed as a percentage:

$$100 \times \frac{Y_{t_1} - Y_1}{Y_1}$$

- delay time for return to normal operation, defined by the period for which the relative deviation of the indication does not exceed $2\sigma_R$ (σ_R being the standard deviation of repeatability at C_1).

NOTE — While the values obtained on the criterion "deviation of indication" can be compared from one device to another, the same is not so for the criterion "delay time" which also involves the repeatability of response for each device. This latter criterion is therefore given only for information on the apparatus under consideration.

COMMENTS

1 In certain cases, the test may reveal, either

- a significant break in operation of the instrument; or
- considerable deviations of indication, after a delay period much greater than the response time, and going beyond the limits compatible with the requirements of practical measurement. In this case, the change over time of the indications after each test will be given and the validity of the results will be judged.

2 An extreme range can be defined by examining the test concentration level C_2 in order to determine the maximum level allowing compliance with the requirements of the two criteria considered:

- deviation of indication;
- delay time.

4.5.3 Summary of information to be given

The following shall be given:

- concentrations C_1 and C_2 ;
- number of operations;
- confidence interval of Y_{t_1} and Y_{t_2} ;
- significant deviation of indication, if any;
- delay time, if any.