



**SLOVENSKI STANDARD**  
**SIST CR 10321:2003**

**01-november-2003**

---

Chemical analysis of ferrous materials - Recommendations for the drafting of standard methods of analysis employing flame atomic absorption spectrometry for the chemical analysis of iron and steel

Chemical analysis of ferrous materials - Recommendations for the drafting of standard methods of analysis employing flame atomic absorption spectrometry for the chemical analysis of iron and steel

**iTeh STANDARD PREVIEW**

Chemische Analyse von Eisenwerkstoffen - Empfehlungen für die Entwicklung von Standard-Analyseverfahren für die chemische Analyse von Eisen und Stahl unter der Anwendung der Flammen-Atomabsorptionsspektrometrie

[SIST CR 10321:2003](#)

<https://standards.iteh.ai/catalog/standards/sist/f556b962-1059-400b-9ddc-10321-2003>

Analyse chimique des matériaux sidérurgiques - Recommendations pour la rédaction de méthodes d'analyse normalisées employant la spectrométrie d'absorption atomique dans la flamme pour l'analyse chimique des fontes et des aciers

**Ta slovenski standard je istoveten z: CR 10321:2003**

---

**ICS:**

77.040.30      Kemijska analiza kovin      Chemical analysis of metals

**SIST CR 10321:2003**      en

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[SIST CR 10321:2003](#)

<https://standards.iteh.ai/catalog/standards/sist/f556b962-1059-400b-9ddc-c1892c44f49e/sist-cr-10321-2003>

CEN REPORT  
RAPPORT CEN  
CEN BERICHT

**CR 10321**

March 2003

ICS

English version

**Chemical analysis of ferrous materials - Recommendations for the drafting of standard methods of analysis employing flame atomic absorption spectrometry for the chemical analysis of iron and steel**

Analyse chimique des matériaux sidérurgiques -  
Recommandations pour la rédaction de méthodes  
d'analyse normalisées employant la spectrométrie  
d'absorption atomique dans la flamme pour l'analyse  
chimique des fontes et des aciers

Chemische Analyse von Eisenwerkstoffen - Empfehlungen  
für die Entwicklung von Standard-Analyseverfahren für die  
chemische Analyse von Eisen und Stahl unter der  
Anwendung der Flammen-Atomabsorptionsspektrometrie

**iTeh STANDARD PREVIEW**

This CEN Report was approved by CEN on 3 October 2001. It has been drawn up by the Technical Committee ECISS/TC 20.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

<https://standards.iteh.ai/catalog/standards/sist/f556b962-1059-400b-9ddc-c1892c44f49e/sist-cr-10321-2003>



EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

**Management Centre: rue de Stassart, 36 B-1050 Brussels**

## Contents

Foreword.....	3
1 Introduction .....	3
2 Instrumental criteria .....	4
3 Adjustment of atomic absorption spectrometer.....	6
4 Preparation of solutions.....	7
5 Calibration procedure.....	8
6 Interferences.....	9
7 Background correction .....	9
8 Expression of results .....	9
9 Safety .....	10
10 Miscellaneous.....	10
11 Summary of recommendations .....	10
12 Literature.....	11
Annex A (informative) <b>Chemical analysis of ferrous material - Determination of calcium in steels - Flame atomic absorption spectrometric method</b> .....	12
Annex B (informative) <b>Precision data</b> .....	18

<https://standards.iteh.ai/catalog/standards/sist/f556b962-1059-400b-9ddc-c1892c44f49e/sist-cr-10321-2003>

## Foreword

This document (CR 10321:2003) has been prepared by Technical Committee CEN/TC 20 "Methods of chemical analysis of ferrous products", the secretariat of which is held by SIS.

Standard methods of chemical analysis have traditionally been based on the techniques of classical chemistry. Generally, by specification of quantities and purity of reagents, such methods may be described completely and thereafter followed exactly in any laboratory.

Today, however, the bulk of analysis is done by instrumental methods for which different laboratories may use instruments from various manufacturers with different configurations and operational settings. It becomes of pressing importance to consider how such methods may be specified in standard documents with regard to both instrument quality and procedure.

This CEN Report considers these matters for methods employing flame atomic absorption spectrometry and makes recommendations. These prescribe instrument quality in terms of limit of detection, curve linearity and precision. Recommendations concerning the preparation of solutions, the calibration procedure and the expression of results are also included. Methods for the determination of calcium in steel and for cobalt in iron steel are included as illustrative examples of the drafting recommendations.

NOTE - Attention is also drawn to CR 10322, Chemical analysis of ferrous materials - Operational guidelines for the application of flame atomic absorption spectrometry in standard methods for the chemical analysis of iron and steel.

INTERNATIONAL STANDARD PREVIEW  
(standards.iteh.ai)

[SIST CR 10321:2003](https://standards.iteh.ai/catalog/standards/sist/556b962-1059-400b-9ddc-c1892c44f49e/sist-cr-10321-2003)

## 1 Introduction <https://standards.iteh.ai/catalog/standards/sist/556b962-1059-400b-9ddc-c1892c44f49e/sist-cr-10321-2003>

An acceptable analytical procedure has to be written in such a form and in such detail that after faithful execution by a well trained analyst an unquestionable result will be obtained. Neither the individual analyst nor the make of apparatus used should have any influence on the final result. These requirements are particularly stringent in the formulation of standard methods used for reference purposes and for arbitration in cases of dispute.

Traditionally, standard methods of analysis have been based on the techniques of classical chemistry. While technically sound, they do not reflect modern developments such as optical emission spectroscopy, X-Ray fluorescence and atomic absorption spectrometry, now in widespread use. New and revised standard procedures now in the course of preparation will increasingly embody these techniques and it becomes pressingly important to consider how such methods may be specified.

With traditional chemical methods there was little problem. Generally, by specification of quantities and purities of reagents, the methods could be described completely and thereafter followed exactly in any laboratory. With instrumental methods, however, the situation is different. Different makes of instruments may be employed in the various laboratories, with different configurations or operational settings, and it becomes more difficult to specify the procedure precisely. Clearly, the standard should not refer to a particular make of instrument, to the implied exclusion of other satisfactory equipment, nor should it include operational settings or measurement times or solution concentrations which may be appropriate to a given instrument only.

On the other hand, if matters are left in the hands of the individual analyst just to 'follow the manufacturer's instructions' the document can no longer be regarded as a standard nor is there any guarantee that the instrument employed will have a quality of performance suitable for the purpose.

The way forward is for the standard document to specify instrument suitability in terms of fundamental performance criteria such as precision, limit of detection and linearity, according to agreed definitions. Individual manufacturers would then be free to meet these requirements in any way they choose. Also, since even a good instrument may deteriorate, the standard should contain reference to practical procedures for the determination of these criteria, to ensure that the instrument meets the specification and continues to do so during its functional years. Other

**CR 10321:2003 (E)**

essential aspects of the standard include instrument optimization, preparation of the test sample, calibration procedures, calculation of results and, where appropriate, how these should be related to the actual instrument used.

This CEN Report considers these matters in relation to standard methods of analysis employing flame atomic absorption spectrometry and makes recommendations for the drafting of such methods. Non-flame atomic absorption spectrometric methods involving instrumental parameters whose definitions and descriptions require further study are not included in this document. The determination of calcium in steel for which performance data are available is included as an illustrative example of the drafting recommendations. A method for the determination of cobalt in steel covering a wide concentration range has also been included in order to illustrate dilution procedures, optimization for minimum matrix interference and correction procedures where cobalt may be present in the iron used for the calibration solutions.

**2 Instrumental criteria**

In the past, attempts have been made to specify instrument performance in a variety of different ways under such headings as minimum sensitivity, curve linearity and minimum stability. It is important that terms used in standard documents should be in accordance with agreed definitions. The term 'minimum sensitivity', for example, has been used to imply a stipulation that the absorbance of the most concentrated calibration solution measured in a flame path length of 10 cm must be at least 0,3. Useful as this requirement may be as a practical guide, it is not the accepted definition of sensitivity in instrumental analysis which is, more properly, the change in instrument reading for unit change in concentration ( $dx/dc$ ), or the slope of the analytical curve at a given concentration. In atomic absorption spectroscopy, the term 'sensitivity' has also been used very widely in manufacturers' literature and elsewhere to imply the concentration of the analyte which will produce a change in the absorbance reading, compared with that of pure solvent, of 0,0044, i.e. 1% net absorption. The IUPAC Commission has drawn attention to this misuse of the term sensitivity and while recognizing the usefulness of the value it represents, has suggested replacement of the name by the term characteristic concentration.

This document recommends that instrument suitability as specified in a given standard document should be based on three performance criteria, according to agreed definitions – limit of detection, acceptable curvature of the calibration graph and precision.

**2.1 Limit of detection**

The limit of detection is smallest concentration in solution of the element of interest which may be detected with confidence. It is obtained from the calibration graph and is generally taken in instrumental analysis as that value on the concentration axis which corresponds to an instrument reading of two or three times the standard deviation above the mean reading of the blank (analytical background).

The IUPAC proposal recommends that this background measurement should be derived from a sufficient number of replicate determinations on the blank solution. Purely to facilitate the practical measurements, the present report suggests a minor modification, i.e. that the measurement should be based on a concentration value selected to give an absorbance of just above the zero and that the standard deviation should be calculated on the basis of 10 replicate determinations. In taking these measurements, as with all other measurements, it is very important that the instrument should be fully optimized. It is not sufficient that it should be adjusted merely to meet the detection limit quoted in the standard. Instrument optimization is considered in some detail in CR 10322.

It is not unusual for the experimentally derived limit of detection to be a few times higher than that quoted by the manufacturer because the latter will have been derived, in all probability, from a new instrument under particularly carefully controlled conditions using pure solutions. If the discrepancy is large, however, the instrument should be overhauled.

The value placed on this detection limit in the standard should be specified with reference to the lowest concentration of the element of interest likely to be encountered in the application envisaged. In the ideal case, the specified limit of detection should be less than one-tenth of the lowest concentration level to be determined and should be measured in the same matrix. The analysis of drinking water which is subject to legal requirements provides a convenient example of this doctrine. If, for example, the maximum legally permitted concentration of lead in potable water were, say, 0,05 ppm Pb (mg/l) and a result very close to this were obtained, it would be necessary to know what confidence could be placed on the result. It would be little comfort to the analyst to feel

that his instrument was working at the very limit of its capabilities. Since the limit of detection of many flame atomic absorption spectrometers is only of the order of 0,01 to 0,02 mg/l Pb, special techniques would be required to give the necessary confidence when operating near the statutory limit. In the case of manganese in potable water, on the other hand, for which the maximum permitted concentration is of the same order, the flame atomic absorption spectrometric technique is very sensitive (limit of detection typically 0,005 mg/l or better), thus meeting the criterion mentioned above and being eminently suitable for direct application.

It is of course recognized that there may be situations where flame atomic absorption spectrometry might still be the method of choice even though the above requirements could not be met. Such situations might exist where the technology does not require residual element determination with a high degree of precision, and a much higher limit of detection relative to the concentration being determined would be acceptable. In this sense, each analytical requirement could be considered on its own merits. Nevertheless, for a standard referee method, as distinct from a routine procedure, specification of a limit of detection of less than one-tenth of the lowest concentration to be determined is a useful guideline.

The exception would be if, despite not meeting this requirement, the method were still the most accurate one available.

## 2.2 Curvature of the calibration graph

It is widely accepted in instrumental analysis that the ideal analytical curve is one which is linear throughout the concentration range of interest. Not only does this situation imply that the measurement system is showing its highest sensitivity but the graph is easy to locate accurately either by a line drawn manually through all the points or by a simple linear 'least squares' mathematical treatment. Most practising analysts feel instinctively that best results are obtained under these conditions.

In practice, however, non-linear calibration graphs are commonplace in atomic absorption spectrometry, even though the inherent curvature may in some cases be concealed by electronic manipulation of the signal before display, i.e. with inherently slightly curved calibration graphs there is the risk that a linear graph is forced through the calibration points which clearly introduces errors. Potential risks associated with a heavily curved region of calibration are that absorbance measurements are insensitive and there may be problems in defining the shape of the curve on the basis of relatively few points in the curved region. It is therefore necessary to consider what constraints should be written into the standard to safeguard analytical accuracy under the conditions on non-linear calibrations graphs.

It has been proposed that the slope of the calibration curve covering the top 20 % concentration range (expressed as a change in absorbance) shall not be less than 0,7 of the value of the slope for the bottom 20 % concentration range determined in the same way. It is recognized that this criterion is not a fundamental requirement in the same sense as an adequate precision. It is quite possible that an instrument just failing to comply with the maximum permitted curvature requirement would give a satisfactory performance if the precision were adequate. Indeed, precision, being a function of sensitivity, curvature, and signal to noise ratio, and the basis on which limit of detection is calculated, is a fundamental performance criterion which, in effect, renders the others redundant. Ringbom plots have shown that acceptable precision can be obtained at absorption values considerably greater than those commonly recommended. Detailed study of such plots enable an analyst to decide on a working range for his instrument which will be appropriate to the precision requirements of his particular application.

However, bearing in mind that accuracy presupposes not only an adequate precision but also freedom from bias and likewise bearing in mind possible difficulties in locating exactly the position of a curved graph, i.e. where departure from linearity becomes significant and how the ensuring shape may be accurately described manually or mathematically in the absence of an inordinately large number of calibration solutions, it is recommended that the above requirement defining the maximum permitted curvature be incorporated in the standard as a guideline.

It is also recommended that information on curvature of the calibration graph should be available for inspection at a point before data processing takes place. While modern developments are welcomed, it is important to ensure that curve straighteners and other electronic devices should not be operating on poor quality input information such as a near horizontal part of the curve, with possibly misleading results of the less experienced analyst.

Recent work has shown that if curvature is due to stray light, linearity requirements will be met if the mid-point calibration absorbance does not exceed 0,55 times full scale absorbance. This provides a particularly convenient and practical way of judging an acceptable linearity.



**CR 10321:2003 (E)****2.3 Precision**

As mentioned in previous clauses, precision is a performance criterion of fundamental significance. A value of precision stated in the standard provides a requirement which proposed instrumentation must meet if it is to be considered suitable for operation of the standard. It provides an assurance against inadvertent use of poor quality equipment.

In some methods, the acceptable precision of measurement at the two ends of the calibration curve has been defined as a stated proportion of the standard deviation of the absorbance of the most concentrated calibration solution.

Typical wording is 'the standard deviation of the absorbance of the most concentrated calibration solution, and the standard deviation of the absorbance of the zero calibration solution, each being calculated from a sufficient number of repetitive measurements, shall be less than 1,5 % and 0,5 % respectively, of the mean value of the absorbance of the most concentrated calibration solution'.

Although perhaps somewhat obscure at first sight, the recommendation has the advantage of relating the required precision to the concentration being determined. Thus assuming for a moment a linear calibration, the precision called for over the concentration range 0,0 % to 1 % of a given element in the above example would be 0,005 % and 0,015 % respectively or, at the 95 % confidence level ( $2\sigma$ ), 0,01 and 0,03 % respectively. The introduction of precision limits based on absorbance measurements in this way also has the advantage of eliminating poor quality equipment at an early stage. The reproducibility of the entire method in terms of concentration units will, however, be affected by other factors, for example curvature of the calibration graph.

The exact values placed on these relative precisions (0,5 % and 1,5 % in the example given above) may well vary from element to element or from standard document to standard document. These values must take account of the purpose of the analysis, i.e. the technical or commercial implications, but will be limited ultimately by the capabilities of the instrument.

Obviously, specified instrumental precisions should not be so generous that the method is incapable of yielding the requisite technical information but it would be equally pointless to set limits in excess of requirements which no instrument is capable of meeting. A comparison of these values with what is available by alternative chemical means will be an important factor in assessing the acceptability of the AAS procedures for standard referee purposes. This point may well be illustrated by reference to some earlier work concerned with the determination of low levels of titanium in steel. It was found that with the instruments at that time available, the precision attainable at low concentrations of titanium was so inferior to the performance of an alternative spectrophotometric procedure as to rule out flame atomic absorption spectrometry for referee purposes, in this particular case.

Two modifications to the above precision requirements may be recommended. Firstly, the number of replicate measurements on which the precision is based should be specified. For consistency with the proposed definition of lower limit of detection, 10 replicate determinations are recommended. Secondly, it is suggested that the second precision measurement should be taken on the lowest calibration standard, instead of on the zero solution. In this way, distortion of the measurement by instrument noise may be minimized. The calibration procedure recommended in clause 5, taken in conjunction with the above precision requirements provides a warning of instrument drift.

**3 Adjustment of atomic absorption spectrometer**

It is assumed that the standard will specify the type of hollow cathode lamp, the wavelength of measurement, and the type of flame, e.g. air-acetylene or nitrous oxide-acetylene, with appropriate burner. Problems with gas purity have been reported. Impure supplies of acetylene have been encountered in which oily drops formed on the burner and the flame was coloured to the extent that it was impossible to see the 'red feather' of the nitrous oxide-acetylene flame. With a satisfactory supply of gas, the air-acetylene mixture will burn with a clear blue transparent flame. It has not so far been found possible to quantify the acetylene purity in percentage terms but in the UK the stipulation 'phosphine-free' has been found to result in a satisfactory gas. Cylinder acetylene is dissolved in acetone. Manufacturers have warned that other solvents may damage plastic components of some instruments and constitute a safety risk. Manufacturers' instructions must be closely followed. Air quality has also been found variable, ranging from an excessive oxygen content to a gas which would not support combustion. The standard should therefore, include a gas quality specification.



Optimization of instrument settings is of vital importance but is one of the more difficult aspects of the method to specify in the standard document. Lamp current, alignment of lamp and burner, warming up time and adjustment of the nebulizer should be covered by the manufacturer's instructions. Burner height and flame composition, however, may be adjusted in various ways. Commonly, the fuel flow and burner position are adjusted for maximum absorbance while aspirating the calibration solution of highest element content. This is a widely used technique but is not favoured as a general procedure because the increase in absorbance may be due to the flame itself.

A preferred method is to make the adjustments so that the difference in absorbance between the highest calibration solution and the blank is at a maximum. When setting for the determination of elements very near their detection limits, however, a procedure which gives a minimum absorbance while water is being aspirated may be adopted, as it simultaneously ensures that the noise level is at a minimum. An alternative optimization procedure may be adopted to minimize severe matrix interference, if this is expected. Optimization of the instrument is considered further in CR 10322.

#### 4 Preparation of solutions

Methods of chemical analysis traditionally recommend that calibration solutions should be prepared to a series of fixed dilutions. However, there is a danger with this approach that a given instrument may not be being used under optimum conditions. It is possible to envisage, for example, that with the stated concentrations, a highly sensitive instrument could give a reading beyond the acceptable upper limit of curvature or even off scale. A different approach is therefore necessary and the optimum concentration range must be determined experimentally because it will depend upon the characteristics of the instrument used.

It has been stated in the literature that the optimum working range of an atomic absorption spectrometer is from 20 to 125 times the characteristic concentration.<sup>1</sup> Assuming a linear calibration, this corresponds to an absorbance of 0,1 to 0,55. This may be used as a basis for the preparation of sample and calibration solutions as follows:

- (i) Determine the characteristic concentration<sup>1</sup> of the element of interest for the given instrument.
- (ii) Prepare an initial sample solution, ideally to have not more than 1 % salts concentration (in order to avoid problems with burner blockage, although higher concentrations may be permissible with some instruments).

From this point there will, in general be four possibilities:

- (i) The solution is too concentrated to be used directly. In this case proceed as follows:

Test the solution at various approximate dilutions to find the solution which falls near the mid-way of the optimum working range – say – 0,25 to 0,45 absorbance.

- (a) Accurately dilute the sample solution by this factor.
- (b) Prepare a series of five or more calibration solutions containing the principal matrix elements in the same concentration as the diluted sample solution with element concentrations to cover the required range, for example, 0 to 140 times the characteristic concentration value (i.e. 0 to 0,6 absorbance).

The above procedure is intended to give optimum performance for referee analysis as befits a standard method.

In routine batch operation, it is recognized that it would be more convenient to make additions to the diluted sample solutions in order to match a predetermined matrix used in preparation of the calibration solutions.

- (ii) The solution is already within the working range of the instrument. In this case dilution is obviously not required and the calibration solutions should be prepared directly as in paragraph (i).

---

<sup>1</sup> N.B. Characteristic concentration, formerly termed 'sensitivity' in atomic absorption spectrometry. See clause 2.

**CR 10321:2003 (E)**

- (iii) The solution is somewhat below the optimum working range of the instrument but cannot be concentrated without causing burner blockage problems or introducing unacceptable separation stages. In this case, an approximate estimation of the element concentration should be made first. Calibration solutions should then be prepared having a similar matrix but with element concentrations of 0, 0,5, 1,0, 1,5, and 2 times the first estimate, or some other suitable multiples.
- (iv) The element concentration is very low indeed, near the detection limit, and cannot be concentrated. If the procedure in paragraph (iii) were followed, the concentration of the highest calibration solution may not be sufficient to give adequate precision. In this case, a range of calibration solutions should be prepared such that the most concentrated will contain sufficient of the element to give full scale deflection when using the maximum scale expansion which is compatible with an acceptable noise level.

**5 Calibration procedure**

A recommended procedure is to aspirate the calibration solutions in ascending order repetitively until each gives the specified precision, thus showing that the instrument has attained stability. Two calibration solutions are then selected, one of absorbance just lower than the sample and one just higher. These are aspirated first in ascending and then in descending order of concentration, with the sample as the middle solution in each case. The complete range of calibration solutions is then aspirated again.

A widespread practice is to obtain the net absorbance of each calibration solution by subtraction of the mean absorbance of the zero calibration solution. At a later stage, a similar procedure involving subtraction of absorbances is followed for the blank and test solutions. When calibration graphs are curved, however, this procedure can introduce significant error. Strictly speaking, absorbances should be converted to concentration values before subtraction, both in the case of the calibration and the test solutions. It is the means of the last complete ascending series of calibration solutions and the descending series of calibration solutions which are used for the calibration graph.

(standards.iteh.ai)

A suggested procedure is to calculate the concentration of analyte in the calibration zero solution using the formula

$$C_z = C_1 \times \frac{A_z}{A_1 - A_z}$$

SIST CR 10321:2003  
<https://standards.iteh.ai/catalog/standards/sist/556b962-1059-400b-9ddc-c1892c44f49e/sist-cr-10321-2003>

where

$C_z$  = concentration of analyte in the zero calibration solution

$C_1$  = concentration of analyte added to the first calibration solution

$A_z$  = absorbance of the zero calibration solution

$A_1$  = absorbance of the first calibration solution

The derived value  $C_z$  is then added to each of the nominal calibration concentration in order to obtain a mean calibration curve passing through the origin. Using this calibration curve, determine the concentration of analyte in the blank test solution for correction of the concentration in the test solution.

The above procedure compensates for the presence of small amounts of the element being determined unavoidably present in the base metal, although high purity metals should always be used where possible.

There are certain circumstances where this relatively lengthy procedure may not be required and absorbances may be subtracted directly without introducing significant error. This situation arises when absorbances of the calibration zero and the blank test are substantially the same, and errors in the operation cancel out.

If results for the two adjacent calibration solution do not deviate from the graph by more than the permitted precision, then the sample results are acceptable. Unacceptable instrumental drift will manifest in failure to meet the precision requirement of clause 2.3.

It is recognized that the above procedures cannot be followed exactly with automatic instruments which accept two calibration solutions only when used in the concentration read-out mode. For this mode of operation, it is first necessary to establish that results obtained in this way would be in agreement with those obtained when the instrument is used in the absorbance mode using carefully drawn calibration graphs based on at least five calibration solutions. Having established this agreement, it is suggested that the two 'sandwiching' solutions should not be used for the primary calibration but should be analysed alternately with the sample. If, however, the validity of the concentration mode of operation cannot be proved, the absorbance read-out with a calibration graph has to be used.

## 6 Interferences

When the method is being tested, all possible interferences from elements in the material being analysed should be investigated at the optimum settings for the instrument and to either side of these settings. In most cases, chemical and physical effects which may influence the atom formation should be largely compensated for by accurate matching of the sample and calibration solution matrices, but if the interference is large, procedures for eliminating it should be written into the method. Such procedures may include addition of reagents to swamp the interference, removal of the interfering element or adjustment of flame composition and burner height to minimize the effect. Since instances have been known where an interference is peculiar to a particular instrument and not to others, the standard should advise the user to test for these effects on his own instrument the first time at which that type of sample is analysed. A simple test would be to add a known quantity of the element being determined to a portion of the sample and to check for recovery. It may be noted that in this CEN Report, there has been no reference to instrumental resolution. This is because true spectral line interference is extremely rare in atomic absorption spectrometry.

# iTeh STANDARD PREVIEW

(standards.iteh.ai)

## 7 Background correction

If, in preparing the initial calibration, the zero calibration solution gives an absorbance comparable with the precision of the lowest calibration solution, background correction will be required. In developing the analytical procedure, typical sample matrices should be checked for non specific absorption. When high concentrations of iron or complex alloys are present, difficulties may be experienced in background correction when using a hydrogen or deuterium lamp because of the complexity and structured nature of the spectral background. The presence of and ways of minimizing such effects, e.g. by selection of spectral band width, should be investigated during development of the method. More modern ways of background correction are now available such as the Zeeman correction or the Smith-Hieftje procedure. The manner of this correction should be according to the manufacturer's recommendations.

## 8 Expression of results

All standard procedures should be supported by performance data. In the ideal case, a minimum of 15 laboratories should collaborate in the testing of the method, with a minimum of three results from each, two under repeatability conditions. Repeatability conditions imply the same operator, apparatus, solutions and calibration, the determinations being done in rapid succession on the same day. In order to obtain a reliable estimate of the standard deviation it is important that the results from all operators should be quoted to the same number of significant figures and, to avoid 'rounding off errors in the statistical treatment, this should be one significant figure more than would normally be quoted in the final result.

Reported results are then subjected to an Analysis of Variance procedure which enables Repeatability,  $r$ , and Reproducibility,  $R$ , to be quoted in the standard document. The reasoning behind these recommendations and worked examples are given in, for example, ISO 5725-2.