
Atomic absorption spectrometer systems for measuring
metal pollutants

Part 2: Test procedures

Systèmes de spectromètres d'absorption atomique pour la mesure des polluants
métalliques

Partie 2: Procédures d'essais



Contents

	Foreword.....	4
1	Description	5
2	Purpose	5
3	Test procedure for flame atomization.....	5
3.1	Equipment	5
3.2	Characteristic concentration.....	5
3.3	Repeatability	6
3.4	IDL	6
3.5	Working range.....	6
4	Test procedure for furnace atomization.....	7
4.1	Equipment	7
4.2	Characteristic mass.....	7
4.3	Repeatability	8
4.4	IDL	8
4.5	Working range.....	8

Foreword

The International Organization of Legal Metrology (OIML) is a worldwide, intergovernmental organization whose primary aim is to harmonize the regulations and metrological controls applied by the national metrological services, or related organizations, of its Member States. The main categories of OIML publications are:

- **International Recommendations (OIML R)**, which are model regulations that establish the metrological characteristics required of certain measuring instruments and which specify methods and equipment for checking their conformity. OIML Member States shall implement these Recommendations to the greatest possible extent;
- **International Documents (OIML D)**, which are informative in nature and which are intended to harmonize and improve work in the field of legal metrology;
- **International Guides (OIML G)**, which are also informative in nature and which are intended to give guidelines for the application of certain requirements to legal metrology;
- **International Basic Publications (OIML B)**, which define the operating rules of the various OIML structures and systems; and

OIML Draft Recommendations, Documents and Guides are developed by Project Groups linked to Technical Committees or Subcommittees which comprise representatives from OIML Member States. Certain international and regional institutions also participate on a consultation basis. Cooperative agreements have been established between the OIML and certain institutions, such as ISO and the IEC, with the objective of avoiding contradictory requirements. Consequently, manufacturers and users of measuring instruments, test laboratories, etc. may simultaneously apply OIML publications and those of other institutions.

International Recommendations, Documents, Guides and Basic Publications are published in English (E) and translated into French (F) and are subject to periodic revision.

Additionally, the OIML publishes or participates in the publication of **Vocabularies (OIML V)** and periodically commissions legal metrology experts to write **Expert Reports (OIML E)**. Expert Reports are intended to provide information and advice, and are written solely from the viewpoint of their author, without the involvement of a Technical Committee or Subcommittee, nor that of the CIML. Thus, they do not necessarily represent the views of the OIML.

This publication – reference OIML R 100-2:2013 (E) – was developed by OIML Technical Subcommittee TC 16/SC 2/p 2 *Water pollution*. It was approved for final publication by the International Committee of Legal Metrology at its 48th meeting in Ho Chi Minh City, Viet Nam, in October 2013 and will be submitted to the International Conference on Legal Metrology in 2016 for formal sanction. OIML R 100 is published in three parts: Part 1: Metrological and technical requirements, Part 2: Test procedures and Part 3 Test report format.

OIML Publications may be downloaded from the OIML web site in the form of PDF files. Additional information on OIML Publications may be obtained from the Organization's headquarters:

Bureau International de Métrologie Légale
11, rue Turgot - 75009 Paris - France
Telephone: 33 (0)1 48 78 12 82
Fax: 33 (0)1 42 82 17 27
E-mail: biml@oiml.org
Internet: www.oiml.org

Atomic absorption spectrometer systems for measuring metal pollutants.

Part 2: Test procedures (Mandatory)

1 Description

This part of R 100 provides test procedures which involve measuring a trace amount of copper in a water solution.

2 Purpose

The purpose of this test is to provide a performance test for an entire AAS system. It covers requirements for an AAS system when using either flame or furnace atomization of the sample. The test results should be consistent with the metrological requirements of clause 5 of R 100-1:2013.

3 Test procedure for flame atomization

3.1 Equipment

Equipment used shall include the following:

- a) copper hollow cathode lamp;
- b) copper reference standard solutions in 0.1 % HNO₃; and
- c) blank test solution of 0.1 % HNO₃.

Note: For the purposes of this test procedure the blank test solution of 0.1% HNO₃ is used to zero the instrument as well as for IDL determination.

3.2 Characteristic concentration

- 3.2.1 Set up the AAS system for a copper determination using the 324.7 nm wavelength, an air-acetylene flame, and set the instrument's parameters as recommended by the manufacturer in the operating manual. Aspirate the blank test solution into the flame, and set the instrument to zero.
- 3.2.2 Select copper reference solutions for aspiration in the flame with concentrations that will permit the interpolation of the value of concentration that gives an absorbance value of 0.1. Divide this concentration by 22.7 to obtain the characteristic concentration. The results shall meet the requirements of 5.4 of R 100-1:2013.

3.3 Repeatability

- 3.3.1 Set up the instrument in the same manner as indicated in 3.2.1. Make ten successive absorbance measurements of a copper reference standard solution with a concentration of approximately 4 mg/L. Calculate the mean and relative standard deviation (%). Use the following equations:

$$s_r = \frac{s}{x} \times 100 \%$$

where

$$s = \left[\frac{\sum (X_i - \bar{X})^2}{n - 1} \right]^{1/2},$$

X_i is the individual result, and

\bar{X} is the mean result for n repeated measurements.

In addition, calculate the instrument absorbance sensitivity, S , as the concentration of the reference standard solution divided by \bar{X} .

- 3.3.2 The results shall meet the requirements of 5.5 of R 100-1:2013.

3.4 IDL

- 3.4.1 Set up the instrument in the same manner as indicated in 3.2.1. Make ten successive measurements of a blank test solution appropriate for the copper reference standard solution. Calculate the mean, and the standard deviation using the second equation indicated in 3.3.1.
- 3.4.2 Multiply the standard deviation (3.4.1) by 3 and, if the standard deviation was initially expressed in absorbance units by the instrument sensitivity, S , (3.3.1). The results shall meet the requirements of 5.6 of R 100-1:2013.

3.5 Working range

- 3.5.1 Set up the instrument in the same manner as indicated in 3.2.1. Select at least five different concentrations of reference standard solution within the values of the working ranges as specified by the manufacturer including concentrations approximately equal to the upper and lower limits of the working range, as specified in Table 1 of R 100-1:2013. At each concentration, ten successive measurements shall be performed, and the mean value and relative standard deviation shall be calculated. The linear curve shall be fitted to the mean values calculated for at least five different concentrations. Use the following equations to calculate the correlation coefficient, r , from the slope, S , of the curve, the standard deviation of the known concentration values, σ_x , and the standard deviation of the measured absorption values, σ_y , from the curve:

$$r^2 = \left[S \times \frac{\sigma_x}{\sigma_y} \right]^2$$

where

$$S = \frac{n \sum (X_i Y_i) - \sum X_i Y_i}{n \sum x_i^2 - (\sum x_i)^2}$$

$$\sigma_x = \left[\frac{\sum (X_i - \bar{X})^2}{n-1} \right]^{\frac{1}{2}}$$

$$\sigma_y = \left[\frac{\sum (Y_i - \bar{Y})^2}{n-1} \right]^{\frac{1}{2}}$$

- 3.5.2 The correlation coefficient, r , determined for the fitted curve in 3.5.1 shall be such that r^2 is equal to or greater than 0.98.

Note: The correlation coefficient, r , interprets how well the data fits the least-squares regression curve. A perfect fit would be $r = \pm 1$ or $r^2 = 1$.

4 Test procedure for furnace atomization

4.1 Equipment

Equipment shall include the following:

- a) copper hollow cathode lamp;
- b) copper reference standard solutions in 0.1 % HNO₃;
- c) blank reference solution of 0.1 % HNO₃; and
- d) a calibrated micropipette, or automatic sampling device, for delivering the sample.

Note: For the purposes of this test procedure the blank test solution of 0.1 % HNO₃ is used to zero the instrument as well as for IDL determination.

4.2 Characteristic mass

- 4.2.1 Set up the AAS system for a copper determination using the 324.7 nm wavelength and set the instrument's parameters as recommended by the manufacturer in the operating manual. Use a pyrolytically-coated graphite tube or an equivalent in the furnace. Set the furnace operating parameters as recommended by the manufacturer, and set the background correction as necessary.
- 4.2.2 Select copper reference solutions to pipette into the furnace with concentrations that will permit the interpolation of the value of concentration that gives an absorbance value of 0.1. Pipette an equivalent quantity of blank test solution into the furnace and measure the peak height or peak area of the output absorbance. Divide the concentration which would give an absorbance of 0.1 by 22.7 to obtain the characteristic mass. The results shall meet the requirements of 5.4 of R 100-1:2013.

4.3 Repeatability

- 4.3.1 Set up the instrument in the same manner as indicated in 4.2.1. Make ten successive absorbance measurements of a copper reference standard solution with a concentration of approximately 20 µg/L. Calculate the mean and relative standard deviation using same equations as in 3.3.1.

In addition, calculate the instrument absorbance sensitivity, S , as the concentration of the reference standard solution divided by \bar{X} .

- 4.3.2 The results shall meet the requirements of 5.5 of R 100-1:2013.

4.4 IDL

- 4.4.1 Set up the AAS system in the same manner as indicated in 4.2.1. Make ten successive measurements of a blank test solution appropriate for the copper reference standard solution. Calculate the mean and standard deviation using the same equations indicated in 3.3.1.

- 4.4.2 Multiply the standard deviation (4.4.1) by 3 and, if it was initially expressed in absorbance units, multiply by the instrument sensitivity, S , (4.3.1). The results shall meet the requirements of 5.6 of R 100-1:2013.

4.5 Working range

- 4.5.1 Set up the instrument in the same manner as indicated in 4.2.1. Select at least five different concentrations of reference standard solution within the values of the working ranges as specified by the manufacturer including concentrations approximately equal to the upper and lower limits of the working range, as specified in Table 2 of R 100-1:2013. At each concentration, ten successive measurements shall be performed, and the mean value and relative standard deviation shall be calculated. The linear curve shall be fitted to the mean values calculated for at least five different concentrations. Use the equations indicated in 3.5.1.

- 4.5.2 The correlation coefficient, r^2 , determined for the fitted curve in 3.5.1 shall be equal to or greater than 0.95.