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
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Foreword

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

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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, \( r^2 \), from the slope, \( S \), of the curve, the standard deviation of the known concentration values, \( \sigma_x \), and the standard deviation of the measured absorption values, \( \sigma_y \), from the curve:

\[ r^2 = \left( \frac{S}{\sigma_y} \right)^2 \]
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 % HNO\(_3\);
c) blank reference solution of 0.1 % HNO\(_3\); 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 % HNO\(_3\) 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 3.3.1.

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.