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QUALIFICATION OF ATOMIC ABSORPTION / ATOMIC EMISSION SPECTROMETERS

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ANNEX 10 OF THE OMCL NETWORK GUIDELINE "QUALIFICATION OF EQUIPMENT"

QUALIFICATION OF ATOMIC ABSORPTION / ATOMIC EMISSION SPECTROMETERS

Introduction

The present document is the tenth Annex of the core document "Qualification of Equipment", and it should be used in combination with it when planning, performing and documenting the Atomic absorption (AA) spectrometer/ Atomic emission (AE) spectrometer qualification process. For AE spectrometer only atomization in flame is considered (Inductively coupled plasma-atomic emission spectrometry is not covered by this guideline).

The core document contains the Introduction and general forms for Level I and II of qualification, which are common to all type of instruments and the present annex contains instrument-related recommendations on parameters to be checked at Level III and IV of qualification and the corresponding typical acceptance limits, as well as practical examples on the methodology that can be used to carry out these checks. The frequency of performing the checks should be defined by each OMCL.

TABLE I

Level III. Periodic and motivated instrument checks

Examples of requirements for AA/AE spectrometers

Parameter to be checked	Typical tolerance limits*
1. Absorption sensitivity	
Flame Test	≥ 0.25 Absorbance units
Furnace test	≥ 0.15 Absorbance units or ±20% of Cu characteristic mass
Hydride vapor generator test	≥ 0.25 Absorbance units
2. Absorption precision	
Flame Test	RSD ≤ 2.0 %
Furnace test	RSD ≤ 2.5 %
Hydride vapor generator test	RSD ≤ 5.0 %
3. Absorption linearity	Correlation coefficient ≥ 0.995
4. Wavelength accuracy	± 0.5 nm
5. Photometric noise	
Background correction Off	≤ 0.01 Absorbance units SD ≤ 0.001
Background correction On	≤ 0.015 Absorbance units SD ≤ 0.001
6. Flame Emission (if applicable) Gain	 ≤ 90 %

* The tolerance limits may be optimized according to the manufacturer's instructions.

TABLE II

Level IV. In-use instrument checks

Examples of requirements for AA/AE spectrometers

Parameter to be checked	Typical tolerance limits*
1. Absorption sensitivity	
Flame Test	≥ 0.25 Absorbance units
Furnace test	≥ 0.15 Absorbance units
Hydride vapor generator test	≥ 0.25 Absorbance units
2. Linearity	Correlation coefficient ≥ 0.99

* The tolerance limits may be optimized according to the manufacturer's instructions.

All parameters given should be checked according to the procedures described in Level III, or alternatively according to Ph. Eur., system suitability requirements in MAH dossier or validated in-house method.

ANNEX I

Level III. Periodic and motivated instrument checks

This Annex contains practical examples of tests and their associated tolerance limits for several parameters related to the Level III qualification of AA/AE spectrometers.

1. Absorption Sensitivity and Precision

The purpose of this test is to determine the absorption sensitivity and absorption precision of the instrument using a reference solution of a known element with a known concentration (certified reference material should be used), for example: copper for flame instruments and manganese or copper for furnace instruments.

Flame test

Reference solutions:
2 mg copper/L water *R* or 5 mg copper/L in water *R*, depending of the instrument characteristics

Method:
Measure the absorbance of the specific element six times in reference solution at the specific wavelength (324.8 nm for copper) according to the manufacturer instructions, and record the values using the appropriate data system.

Limits:
The test passes if the absorbance reading is within the expected value (≥ 0.2 Absorbance units for chosen concentration) and the value for relative standard deviation (% RSD) measured absorbance of the consecutive measurements is ≤ 2.0 %.

In alternative, the absorbance reading is within ± 20% of absorbance value specified by the method provided by the instrument manufacturer.

Furnace test

Procedure 1:

Reference solution:
1 μg manganese/L water *R* or 25 μg copper/L in water *R* or other appropriate element at the given concentration, according to instrument manufacturer instruction.

Method:
Measure the absorbance of the specific element six times in reference solution at the specific wavelength (279.5 nm for manganese or 324.8 nm for copper) according to the manufacturer instructions and record the value using the appropriate data system.

Limits:
The test passes if the absorbance reading is within the expected value (≥ 0.15 Absorbance units) and the value for relative standard deviation (RSD %) measured absorbance of the consecutive measurements is ≤ 2.5 %.

This limit is applicable using manganese or copper reference solution prepared as described above (Procedure 1), when other elements are used the limit is to be adjusted taking into consideration the manufacturer's specification.

Procedure 2:

Prepare a copper reference solution and measure the characteristic mass as described by the manufacturer.

Limits:
± 20% of copper characteristic mass, as specified by the manufacturer.

Hydride vapor generator test

Reference solution:
Prepare a solution containing 10 μg arsenic/L in water *R* containing 2 mL hydrochloric acid *R* and 2 mL of 20 % v/v potassium iodide.

Method:
Measure the absorbance of the specific element six times in reference solution (10 μg arsenic/L) at the specific wavelength (193.7 nm for arsenic) according to the manufacturer instructions, and record the value using the appropriate data system.

Limits:
The test passes if the absorbance reading is within the expected value (≥ 0.25 Absorbance units) and the value for relative standard deviation (% RSD) measured absorbance of the consecutive measurements is ≤ 5 %.

2. Absorption Linearity

The purpose of this test is to determine the linear relationship between the measured absorbance and the concentration of the multiple certified standard solutions of a known element (for example, copper for flame instruments and manganese or copper for furnace instruments).

Reference solution:
Prepare at least three reference solutions of copper for flame instruments and manganese or copper for furnace instruments over the calibration range (depending of the instrument characteristics) and a blank solution.
For example:
For flame instruments:
- Reference solutions of copper: 0 μg/mL, 1 μg/mL, 2.5 μg/mL, 3.5 μg/mL and 5 μg/mL
For furnace instruments:
- Reference solutions of copper: 0 μg/L, 10 μg/L, 15 μg/L, 20 μg/L and 25 μg/L
- Reference solutions of manganese: 0 μg/L, 0.1 μg/L, 0.5 μg/L, 1.2 μg/L and 2.0 μg/L
Hydride vapor generator
- standard solutions of arsenic: 0 μg/L, 2.5 μg/ L, 5 μg/ L, 7.5 μg/ L and 10 μg/ L
Solvent: water *R*.

Method:
Measure the absorbance of the specific element in standard solutions (at least three replicates for each concentration) at the specific wavelength (279.5 nm for manganese or 324.8 nm for copper) according to the manufacturer instructions, and record the values using the appropriate data system. Calculate the calibration curve by least-square regression from all measured data.

Limits:
The test passes if the correlation coefficient is at least 0.995. Residuals of each calibration level are randomly distributed around the calibration curve.

For furnace instruments with autosampler:

If an auto sampler is used, which mixes the calibration solutions automatically, the autosampler linearity should be checked instead of absorption linearity.

Limits:
The test passes if the correlation coefficient is at least 0.995.

3. Wavelength accuracy

Method:
The accuracy of the wavelength can be verified by measuring the emission lines of a certified hollow cathode lamp for the elements at the low, middle and high end of the spectrum. For example: As (193.7 nm), Cu (324.8 nm), Na (585.3 nm) and K (766.5 nm).
Alternatively, if the specified hollow cathode lamps are not available, verify the accuracy of the wavelength readings by measuring the emission lines of an Hg hollow cathode lamp (for example: 253.6 nm, 365.0 nm, 435.8 nm, 546.1 nm, 585.5 nm, 640.2 nm and 724.5 nm).

Limits:
The test passes if the measured values are within ± 0.5 nm of the wavelengths of the used lamp.

4. Photometric noise

Method:
The test is performed without using any references solution.
Read the changes in absorbance within a set period of time (period is set according to manufacturer's instructions) around 0 Absorption units in the non background correction mode (BC off) and then in the background correction mode (BC on). The absorbance fluctuation is taken as the BC off noise level and BC on noise level. The test is performed according to the instrument's instruction manual.
Depending on the instrument type, the test for the BC off mode may be performed with additional reading of the changes in absorbance within a set period of time (period is set according to manufacturer's instructions) around 1 Absorption units in the non background correction mode (BC off).

Limits:
The test passes if the measured values for the noise level are $\leq$ 0.01 Absorbance units for the BC off mode and $\leq$ 0.015 Absorbance units for the BC on mode.
The standard deviation of the mean result in integrated absorbance must not exceed 0.001.

Other approaches/limits described by manufacturers may be used.

5. Flame Emission (automated software test, if applicable).

Limits:
Apply the limits as set up by manufacturer.