INTERNATIONAL
RECOMMENDATION

OIML R 100-1

Edition 2013 (E)

Atomic absorption spectrometer systems for measuring metal pollutants

Part 1: Metrological and technical requirements

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

Partie 1: Exigences métrologiques et techniques

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INTERNATIONAL ORGANIZATION
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Foreword

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This publication – reference OIML R 100-1:2013 (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.

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Atomic absorption spectrometer systems for measuring metal pollutants.

Part 1: Metrological and technical requirements

1 Scope

1.1 This Recommendation provides requirements for defining, testing and verifying the performance of atomic absorption spectrometer (AAS) systems when used for measuring metal pollutants in water as mandated by national laws and regulations. It does not intend to exclude any other equivalent means of measuring metal pollutants in water. An AAS system can be applied simply and rapidly for the determination of a variety of metals in potable, ground, surface and saline waters as well as domestic and industrial wastewaters. An AAS system of this type may be applied for the determination of metal concentrations in other matrices such as soil, etc. References [1] – [4] give a description of the AAS system and some applications.

1.2 The flame and electrothermal atomization (furnace) types of AAS atomizers are covered by this Recommendation. The hydride generation and the cold vapor techniques are also briefly described. The conditions of operation described for each system type are intended to cover applications for trace analysis.

1.3 It is possible that the performance of an instrument can exceed the minimum criteria prescribed in this Recommendation for these applications. Higher accuracy and repeatability of measurements can be accomplished by careful attention to the optimization of the performance of each of the instrument’s components. In such cases, success may also depend on the knowledge, skill, and experience of the analyst, as well as the quality of instrument manufacture.

1.4 A sample usually needs to undergo a specific preparation technique prior to analysis with an AAS system. Sampling techniques, methods of analysis and impact of interfering components are beyond the scope of this Recommendation. As appropriate, the relevant international, regional or national sampling techniques and reference methods of analysis should be applied.

1.5 This Recommendation does not address requirements and tests that may be necessary for operator safety. Therefore, users should determine whether an instrument meets the safety and labeling requirements specified in national regulations.

2 Application

2.1 This Recommendation applies to AAS systems used for measuring metal pollutants in water, in assessing water quality, and as a means of monitoring the effectiveness of pollution control programs mandated by national laws or regulations. Such control programs are usually designed to assess whether the concentration of metals is within regulated limits for toxic and non-toxic pollutants in potable, surface, ground and saline waters.

2.2 Normally, after simple acidification, water free of particulate matter may be analyzed directly by AAS systems. Domestic and industrial wastes shall in general require processing to dissolve suspended material, in line with appropriate standard analysis methods. Sludge, sediments and other types of solid samples may also be analyzed after proper digestion and pretreatment.

3 Terms and definitions (Terminology)

References [5] and [6] provide definitions of terms relevant to this Recommendation. The definitions of some terms presented here, however, have been modified for better interpretation of this application.

3.1 absorbance

logarithm of the ratio of incident to transmitted radiant power, which is dependent on the path length and the concentration of the absorbing substance

3.2 atomizer

device for converting the analyte into atomic vapor

3.3 blank solution

3.3.1 blank reference solution

solution used to set the zero absorbance on the spectrometer and that normally consists of a pure solvent such as de-ionized water

3.3.2 blank test solution

solution that contains all the chemicals except for the element to be determined in the same concentration as required for the preparation of a reference standard solution of that element

3.4 reference standard solution

solution containing an accurately known concentration of a sample element or elements of interest and that is used for testing and calibrating the instrument

3.5 characteristic concentration/characteristic mass

concentration or mass of an element which produces a change from the blank test solution of 0.004 4 absorbance units (1 % absorption) at the wavelength of the absorption line employed

Note: Historically, sensitivity has been used for this term in this field.

3.6 instrument detection limit (IDL)

figure of merit which is calculated as three times the observed standard deviation of the baseline noise, established by measuring the output signal from a blank test solution free of interferents

3.7 working range

range of concentrations of an element of interest in solution that can be measured within specified limits

Note: Dilution, or concentration, of a sample solution may be necessary to bring the element of interest within the working range. The zero absorbance of the spectrometer is adjusted using a blank reference solution.

3.8 nebulizer

device that converts a liquid sample into an aerosol

3.9 repeatability

closeness of agreement between the results of successive measurements of the same quantity being measured using the same instrument and under the same defined conditions within a relatively short period of time

3.10 temperature programming

means of automatically controlling the rate and duration of the temperature rise of a furnace-type AAS system

4 Description of the instrument

4.1 General

Atomic absorption spectrometry is based on the absorption by an atom in its ground state of characteristic resonance radiation produced by atoms of the same element contained in a light source. Light absorbed by the atomized sample is measured and can be related to the concentration of the element in the sample. In flame atomization, a steady-state absorption signal is normally measured while in furnace atomization, a measurement is made of either the peak height or peak area of a transient signal. Physical and chemical interferences with the light absorbed by the element can occur that are unrelated to the element's concentration. A number of techniques may be used to eliminate or minimize such possible interference.

4.2 Flame AAS system (F-AAS)

A diagram of the main components of the instrument is given in Figure 1. The components are as follows:

A light source that emits the characteristic narrow-line spectrum of the element of interest;

B flame into which the sample solution to be analyzed is aspirated by the nebulizer (C);

C nebulizer, which, when in the flame, passes through several stages:

- first the liquid solvent is evaporated;
- then the solid sample is vaporized to a gas; and
- then the compounds making up the sample are broken into free atoms by thermal molecular dissociation;

D monochromator for dispersion of the light into its discrete spectral wavelengths that can be selected for analysis with slits of variable width;

E suitable radiation detector; and

F readout system for displaying or storing the output signal and subsequent data processing. Spectral background correction may be necessary for a flame AAS system especially at wavelengths below 300 nm.

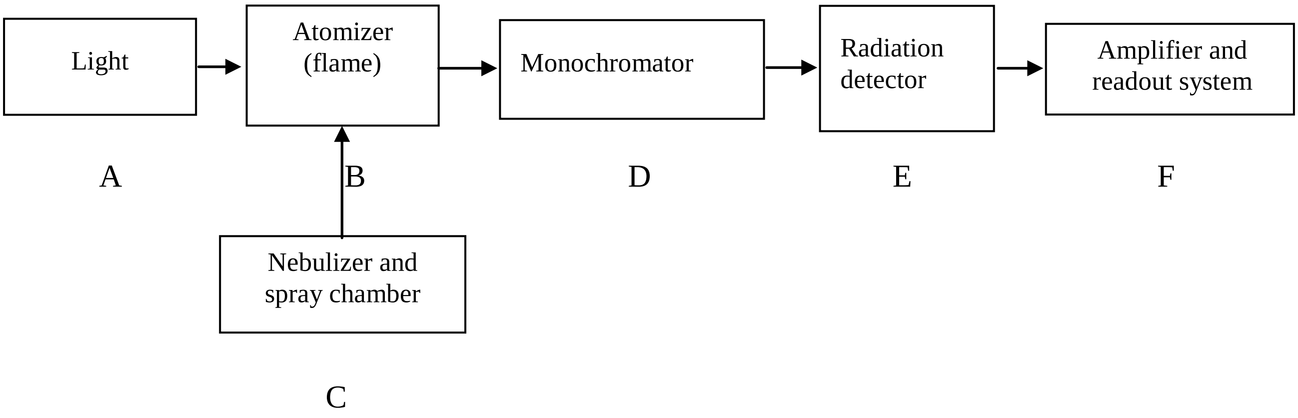

Figure 1 – Diagram of a flame absorption spectrometer

4.3 Furnace AAS system (GF-AAS)

This instrument has basically the same major components as a flame AAS system except for the fact that a furnace, in which the heated element is a graphite tube, replaces the flame. A sample solution is deposited in the furnace either directly on the tube wall or on a graphite platform. In some cases, a matrix modifier may be required. The temperature of the graphite tube is then increased progressively in a programmed manner to dry the sample, char (or ash) the matrix material, and atomize the element. Since this process is subject to non-specific absorption, spectral background correction is necessary. The repeatability of measured results is best when using an automatic sample introduction device.

Note: The IDL of a furnace AAS system is a factor of ten to a thousand times lower than that of a flame AAS system; however, the repeatability of measured results by a flame AAS system is usually better than those of a furnace AAS system. Flame analysis can be performed more quickly than furnace analysis and is less dependent on the matrix of the sample. The sensitivity enhanced detectivity obtained by the furnace technique can decrease with the number of firings (age), and the change in performance may depend on the nature of the graphite tube.

4.4 Other techniques

4.4.1 Hydride AAS system (HG-AAS)

This technique involves the generation of volatile hydrides of the element of interest in the sample solution undergoing analysis. The sample solution, an acid, and a reducing agent are reacted, either in a batch or continuous flow system, and the liberated hydride is transported to an atomizer by an inert carrier gas. The advantage of this technique over flame AAS is the separation and enrichment of the element to be determined and the significantly lower IDL resulting from greater efficiency of sample introduction. This method may have more interference effects than the flame AAS system.

4.4.2 Cold vapor AAS system (CV-AAS)

This instrument applies only to mercury, which can exist in a gaseous mono-atomic state at ambient temperature because of its relatively high vapor pressure. A reducing agent, usually either stannous chloride or sodium borohydride, is mixed with an acidic sample solution containing trace amounts of ionic mercury either in a batch or continuous flow system. The liberated atomic mercury vapor is then transported to an absorption cell by air or an inert gas. This technique for determining mercury is much more sensitive than a flame AAS system.

5 Metrological requirements

5.1 The metrological characteristics required of the flame AAS system and the furnace AAS system are given in Table 1 and Table 2, respectively. Information is also given in Table 3 regarding the metrological characteristics of an AAS system using hydride and cold vapor techniques.

5.2 During type evaluation and initial and subsequent verification, the repeatability, IDL, and working range of the output signal of the instrument shall be tested in a laboratory using reference standard solutions with temperature and relative humidity within the following ranges throughout the characterization:

- ambient temperature of 23 °C ± 3 °C; and
- relative humidity (RH) from 30 % to 80 %.

5.3 The manufacturer shall specify the following components and other conditions under which the instrument is to be tested:

- flame AAS
  - light source;
  - flame fuel and flow rates;
  - atomizer;
  - slit width for the optical system;
- furnace AAS;
  - light source;
  - temperature programming;
  - slit width for the optical system.

5.4 The characteristic concentration of the AAS system shall be checked. The procedure shall be to determine the concentration of a reference standard solution that provides a value of absorbance as defined in 3.5. The result shall be equal to or less than the value for characteristic concentration given for an AAS flame or a furnace system as specified in Tables 1, and 2, or the values specified by the manufacturer of the system. Detailed procedures for these tests can be found in R 100-2:2013.

5.5 The repeatability of measurements shall be checked. The procedure shall include measuring ten repeated samples of the reference standard solution containing the relevant test element with a concentration equivalent to approximately the value at the mid-point of the specified working range as specified in Table 1 or 2 (as appropriate). The observed relative standard deviation of the measurement results shall be less than 2 % for the flame AAS system and less than 5 % for the furnace AAS system. Detailed procedures for these tests can be found in R 100-2:2013.

Note: Recommended concentrations of stock solutions for preparing reference standard solutions are given in Annex A.

5.6 The IDL of a specified element shall be checked by measuring ten repeated samples of a blank test solution at its wavelength of detection. The standard deviation of the measurement results converted to concentration units shall be multiplied by three to give the IDL, which shall be less than or equal to the values given in Tables 1 and 2.

5.7 The working range shall be determined. The procedure shall include fitting a curve to data obtained for at least five different concentrations of a reference standard solution within the values of the working ranges. The concentrations shall include those approximately equal to the values at the upper and lower limits of the working range as specified for the AAS flame and furnace systems in Tables 1 and 2, respectively. At each concentration, ten replicate measurements shall be performed, and the mean value and standard deviation shall be calculated. The linear curve shall be fitted to the mean values calculated for at least five different concentrations. The correlation coefficient, r, of the fitted curve shall be determined, and r^2 shall be equal to or greater than 0.98 for the flame AAS system and 0.95 for the furnace AAS.

Table 1 – Metrological characteristics of a flame AAS system[1]

Element	**Wavelength** nm	**Flame type**[a]	**Characteristic concentration**[b] mg/L	**IDL** mg/L	**Working range** mg/L
Aluminum	309.3	NA	1.0	0.05	10–100
Antimony	217.6	AA	0.4	0.08	4–40
Arsenic	193.7	AA or NA	1.0	0.25	10–100
Barium	553.6	NA	0.5	0.02	5–50
Beryllium	234.9	NA	0.03	0.002	0.3–3
Cadmium	228.8	AA	0.03	0.002	0.3–3
Calcium	422.7	AA or NA	0.1	0.004	1–10
Chromium	357.9	AA or NA	0.08	0.005	0.8–8
Cobalt	240.7	AA	0.12	0.008	1.2–12
Copper	324.7	AA	0.08	0.003	0.8–8
Iron	248.3	AA	0.1	0.006	1–10
Lead	283.3	AA	0.5	0.05	5–50
Lithium	670.8	AA	0.04	0.002	0.4–4
Magnesium	285.2	AA	0.008	0.000 5	0.08–0.8
Molybdenum	313.3	NA	0.7	0.1	7–70
Nickel	232.0	AA	0.15	0.02	1.5–15
Selenium	196.0	AA or NA	0.6	0.2	6–60
Silver	328.1	AA	0.06	0.002	0.6–6
Sodium	589.0	AA	0.015	0.000 8	0.15–1.5
Strontium	460.7	AA or NA	0.16	0.012	1.6–16
Vanadium	318.4	NA	2.0	0.1	20–200
Zinc	213.9	AA	0.02	0.002	0.2–2

[a] The symbols AA and NA represent air-acetylene and nitrous-oxide-acetylene flames, respectively.

[b] Historically called sensitivity. Consult values specified by the instrument manufacturer (if available).

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[1] For certain elements such as sodium or lithium, an ionisation suppressor such as caesium may be necessary in order to increase sensitivity and linearity.

Table 2 – Metrological characteristics[a] of a furnace AAS system

Element	Wavelength nm	Characteristic mass[b] pg	Characteristic concentration[b] μg/L	IDL μg/L	Working range μg/L
Aluminum	309.3	8	0.4	0.2	4–40
Antimony	217.6	22	1.1	0.8	11–110
Arsenic	193.7	20	1	1	10–100
Barium	553.6	8	0.4	0.2	4–40
Beryllium	234.9	2	0.1	0.03	1–10
Cadmium	228.8	0.4	0.02	0.03	0.2–2
Chromium	357.9	8	0.4	0.1	4–40
Cobalt	240.7	16	0.8	0.2	8–80
Copper	324.7	8	0.4	0.2	4–40
Iron	248.3	6	0.3	0.2	3–30
Lead	283.3	24	1.2	0.3	12–120
Magnesium	285.2	0.2	0.01	0.004	0.1–1
Molybdenum	313.3	24	1.2	0.45	12–120
Nickel	232.0	40	2	1	20–200
Selenium	196.0	16	0.8	2.5	8–80
Silver	328.1	1	0.05	0.05	0.5–5
Vanadium	318.4	80	4	4	40–400
Zinc	213.9	0.6	0.03	0.03	0.3–3

[a] A typical sample solution is from 10 μL to 20 μL. Values in this Table are based on the absolute mass and concentration for a sample solution of 20 μL. Therefore, for other volumes of the sample, the values should be adjusted accordingly, for example a sample of 10 μL, all values except characteristic mass would be doubled.

[b] Each value represents an example, consult values specified by the instrument manufacturer (if available).

Table 3 – Metrological characteristics of an AAS system using hydride and cold vapor techniques[a]

Element	Wavelength nm	Characteristic concentration μg/L	IDL μg/L	Working range μg/L
Arsenic	193.7	0.04	0.15	0.4–4
Antimony	217.6	0.08	0.24	0.8–8
Mercury	253.7	0.8	0.2	8–80
Selenium	196.0	0.06	0.24	0.6–6
Tin	286.3	0.4	1.0	4–40

[a] Mercury is to be determined by the cold vapor technique (see [6]). The other elements listed are to be determined by the hydride technique. This Table applies to the determination of elements in sample solutions using a batch mode; however, instruments operating in continuous flow mode may have slightly different metrological characteristics than those presented.

[b] Values in this Table are based on a sample size of 50 mL and for the determination of the elements under optimum conditions. Because of the high sensitivity of the hydride technique, a sample solution of 50 mL is rarely required; sample solutions with volumes of 1 mL or less are generally sufficient to permit analysis within the working range (see [1]).

6 Technical requirements

6.1 Atomizer

6.1.1 The flame atomizer shall be a nebulizer/premix type, in which the liquid sample is sprayed into a mixing chamber where the sample aerosol and the oxidant are mixed with the fuel. Analyses shall be carried out using either an air-acetylene flame for temperatures up to 2 300 °C or nitrous oxide-acetylene flame for temperatures up to 2 900 °C. Table 1 gives recommendations of the flame to be used for the determination of various elements. The manufacturer shall specify the required purity and flow rates of compressed air, acetylene, and nitrous oxide.

Note: For safety reasons, when cylinders of compressed acetylene (ethyne) are shipped and/or stored, the acetylene is always dissolved in acetone or dimethylformide (DMF).

6.1.2 A furnace AAS system shall have a means for achieving temperatures necessary for atomizing the element of interest in a sample. It shall be possible to program various furnace temperatures in a step-wise mode to dry the sample, to remove the matrix material by pyrolysis or ashing, and then to atomize the element of interest. The manufacturer shall specify temperature programming available. A means for spectral background correction shall be available during analyses.

6.2 Light sources and optical system

The manufacturer shall specify the type of and necessary adjustments for the light sources, optical system, and detectors used.

6.3 Measurement capability

The spectrometer shall be capable of measuring wavelengths over the range of at least 190 nm to 770 nm.

6.4 Monochromator

The monochromator shall be equipped with slits that can be pre-selected or adjusted to select the desired bandwidth for the wavelength of the element being determined.

6.5 Data system

The data system shall be capable of producing accurate, archivable, and retrievable records of the detector response and other essential data of analysis.

6.6 Markings

Markings shall be attached conspicuously to all major components of an AAS system as follows:

- name of the manufacturer;
- instrument model, serial number, and date (month and year) of manufacture;
- voltage, frequency, and current requirements.

Note: Labels or declarations concerning personnel safety and radio frequency interference emission should be provided according to national regulations.

6.7 Manuals

The manufacturer of an AAS system shall supply a manual that describes the requirements for its installation, operation, and routine maintenance. Service manuals shall be available upon request.

Note: The manufacturer may provide analytical methods manuals.

7 Practical instructions

7.1 Operation of an AAS system requires the use of high voltages and high temperatures during normal operation. Instrument installation and operation, particularly with respect to flames and compressed gases, shall take into account these requirements. Warning labels shall be placed conspicuously on the instrument to alert users to these potential hazards. Such labels shall be consistent with national safety regulations.

7.2 Before installation of an AAS system, all laboratory environmental factors shall be considered. The manufacturer shall provide specifications for power consumption that include allowable variations from the rated mains voltage and frequency. Information shall also be provided for rated heat dissipation, and operating conditions for ambient temperature and humidity, and exhaust-gas venting.

8 Metrological controls

8.1 Type evaluation

8.1.1 The manufacturer shall provide the responsible national body with the identified AAS system and its operating manual. The manufacturer may also provide data and other information that support a determination of whether the system meets the requirements of this Recommendation.

8.1.2 The national responsible body shall review the system’s operating manual for the completeness and clarity of instructions. The system shall be visually inspected in conjunction with a review of the manufacturer’s specifications to determine whether the requirements of clause 6 are met.

8.1.3 The national responsible body shall carry out tests, or may accept the manufacturer’s test data, to confirm the conformity of the system with the following performance requirements:

- characteristic concentration (5.4, and 3.2 and 4.2 of R 100-2:2013);
- repeatability (5.5, and 3.3 and 4.3 of R 100-2:2013);
- IDLs (5.6, and 3.4 and4.4 of R 100-2:2013);
- working range (5.7, and 3.5 and 4.5 of R 100-2:2013).

8.1.4 The report of tests on the system carried out during type evaluation shall contain, as a minimum, the items of information according to the format specified in R 100-3:2013.

8.2 Initial and subsequent verification

8.2.1 The documentation provided by the manufacturer shall be examined to ensure compliance with 8.1.

8.2.2 The test under reference conditions specified in 5.4 shall be carried out.

8.2.3 Any of the other tests specified for the AAS system under 8.1.3 that are considered critical to the specific application shall be performed.

8.2.4 The national responsible body shall specify the period of validity of the verification.

8.2.5 An instrument shall undergo subsequent verification equivalent to initial verification after repair or replacement of major component parts of the spectrometer and the data handling system or after the period of time specified by the national responsible body.

8.3 Routine tests by the user

8.3.1 The national responsible body shall identify methods for using AAS systems to measure specific pollutants. Some measurement methods may be appropriate for use in assessing the performance of an AAS system.

8.3.2 The operator of an AAS system shall carry out a test with a reference standard solution before and after a series of measurements in the laboratory. The results of these tests shall be within the limits established by the national responsible body.

8.3.3 In addition to the requirements of 8.3.2, an operator shall carry out a performance test of the entire AAS system related to and before a specific application.

8.3.4 A chronological record shall be maintained for each AAS system and shall contain at least the following information:

- results of all routine tests;
- results of calibrations;
- identification of any major components replaced;
- details of maintenance and repair.

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Annex A – Reference standard solutions

(Mandatory)

A.1 Preparation of reference standard solutions

A.1.1 A stock solution of each element of interest shall either be obtained from commercial sources or shall be prepared from ultra-high purity grade elements or their salts. These solutions shall be prepared using ultra-high purity grade hydrochloric acid or nitric acid, as appropriate, and shall have an acid concentration such as to produce a stable solution of the element of interest unless otherwise directed by the supplier of the material. The water used in preparation of the stock standard solutions and in subsequent dilutions shall have an electrical conductivity at 25 °C of less than 0.1 mS/m (grade 2 water according to ISO 3696).

Note 1: The stock solutions shall be compared with suitable certified reference materials in order to establish traceability of the calibration material. Stock solutions with known certified values are preferred.

Note 2: Stock standard solutions usually have a concentration of the element or elements of interest of 1 000 mg/L or 10 000 mg/L.

Note 3: Stock standard solutions should be prepared using appropriate and documented standard methods.

Note 4: All salts used as a source for an element should be dried at 105 °C for one hour except for carbonates, which should be dried at 140 °C for one hour.

A.1.2 The concentrations of the reference standard solutions shall be those specified in R 100-2:2013 for the particular test and type of AAS and within the appropriate working ranges of Table 1 or Table 2. Reference standard solutions shall be prepared by appropriate dilution of the stock standard solutions described in A.l.1. The selection of an acid and its concentration shall be made so as to provide compatibility for any mixed standards being prepared.

A.1.3 Reference standard solutions shall be monitored to ensure their stability with time, usually by using quality control charts.

A.2 Preparation of mixed reference standard solutions

A.2.1 Mixed (multi-element) reference standard solutions containing several elements of interest in the same solution may be prepared using suitable combinations and dilutions of the stock standard solutions within the concentration working range in Tables 1 or 2. Before preparing the mixed reference standard solutions, the stock standard solutions to be used shall be analyzed to identify any potential spectral interference at the wavelengths selected for analysis.

A.2.2 The mixed reference standard solutions shall contain elements that are compatible with each other and stable in the mixture. The compatibility of the anions shall also be considered.

Note: Any acids added to preserve the mixed solution should be compatible with all elements included.

A.2.3 The mixed reference standard solutions shall be monitored periodically for stability.