FINAL DRAFT
RECOMMENDATION

TC 16/SC 2/p 2
(USA)

INFORMATION

Final Draft Revision of R 100

Atomic absorption spectrometer systems for measuring metal pollutants in water

Final Draft submitted for CIML online approval
on 2013.04.12.
Voting closes on 2013.07.12.

Final Draft Revision R 100

ORGANISATION INTERNATIONALE
DE MÉTROLOGIE LÉGALE

INTERNATIONAL ORGANIZATION
OF LEGAL METROLOGY

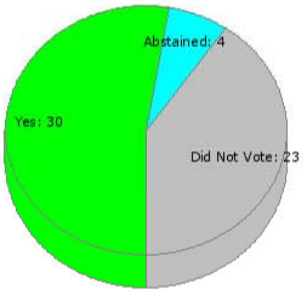
OIML

CIML Preliminary ballot of the draft revision of OIML R 100 "Atomic absorption spectrometer systems for measuring metal pollutants in water"

Deadline: 2012-08-24

AUSTRALIA voted Yes (with comments)
AUSTRIA voted Yes
BELARUS voted Yes
BELGIUM voted Yes
CANADA voted Yes
CYPRUS voted Yes
CZECH REPUBLIC voted Yes
DENMARK voted Yes (with comments)
FINLAND voted Yes
FRANCE voted Yes
GERMANY voted Yes
HUNGARY voted Yes
IRELAND voted Yes
JAPAN voted Yes
KAZAKHSTAN voted Yes
MONACO voted Yes
NEW ZEALAND voted Yes
NORWAY voted Yes
P.R. CHINA voted Yes
POLAND voted Yes
ROMANIA voted Yes
RUSSIAN FEDERATION voted Yes
SERBIA voted Yes
SLOVAKIA voted Yes
SOUTH AFRICA voted Yes
SWEDEN voted Yes
SWITZERLAND voted Yes (with comments)
TANZANIA voted Yes
UNITED STATES voted Yes
VIET NAM voted Yes

INDONESIA Abstained
NETHERLANDS Abstained
SLOVENIA Abstained
UNITED KINGDOM Abstained (with comments)



Countries who did not vote (23)

ALBANIA, ALGERIA, BRAZIL, BULGARIA, CROATIA, CUBA, EGYPT, GREECE, INDIA, IRAN, ISRAEL, ITALY, KENYA, KOREA (R.), MACEDONIA (F.Y.R.), MOROCCO, PAKISTAN, PORTUGAL, SAUDI ARABIA, SPAIN, SRI LANKA, TUNISIA, TURKEY.

Country	Clause	Comment	Response
UK	General	We are pleased to see that a significant number of points raised in our response of 15 September 2010 have been agreed. While we appreciate and understand the reasons for some of the comments not being agreed, there are some which we still strongly believe need to be considered. For this reason we feel we cannot vote for the Recommendation. However, as we do not want to vote against the Recommendation we have decided to abstain.	The Secretariat thanks the UK reviewers on their diligence and helpful comments.
DK	General	The recommendation is superfluous. Very suitable ISO standards are available.	ISO standards are focused on methods of measurement in complex mixtures. The OIML recommendation is focussed on the qualification of instrumentation that will be eventually used to carry out complex chemical quantitation.
CH	2.2, line 4	instead of "…proper pretreatment." ".,. proper digestion and pretreatment."	Agreed, text has been modified in consideration of the comment.
CH	3.1, line 2	use plural: "the concentrations of the absorbing substances."	Can be either singular or plural, text is unchanged.
AU	3.5	Is the blank referred to the 'blank reference solution' or 'blank test solution'?	Blank test solution, text has been modified.
CH	3.6, line 1	Delete "true". The true value concept is outdated since 1993 (cf. ISO-GUM). Therefore calling a "true concentration" is not acceptable.	agreed

AU	3.6	Is the definition at 3.6 in harmony with the IUPAC Compendium of Chemical Terminology (Gold Book) which says the limit of detection is mean of the blank plus the standard deviation (sd) multiplied by a numerical factor chosen according to desired confidence level. In R 100 the zero absorbance of the instrument is adjusted using a blank reference solution (e.g. deionized water) at 3.3.1 (blank reference solution) and 3.7 (working range). If the detection limit is determined using the blank reference solution, then the mean of the blank is treated as zero and the detection limit is just a multiple of the sd. If the detection limit is determined using the blank test solution (aka matrix blank), then the mean of the blank is non-zero. In R 100, the limit of detection is determined using the blank test solution however the mean of the blank is not considered. In addition to the issue of how the limit of detection is calculated, it may be helpful if R 100 used more explicit terminology such as 'instrument detection limit' (IDL) and 'method detection limit' (MDL).	This recommendation uses the instrument detection limit and not the limit of detection. 3.6 has been modified to clarify this confusion. Recommendation now refers to IDL to prevent future confusion The definition is in harmony if the blank reference solution used to zero the instrument is the same as the blank test solution, which is the case in R100.
CH	3.7	"…that can be measured within specified limits." Again trueness is an outdated term, that is no more in use today. In this context the term is superfluous and not related to the definition.	agreed
CH	4.1, line 6,8	use plural: "…chemical interferences", "… possible interferences."	agreed
CH	4.2, title	add accepted abbreviation: Flame AAS system (F-AAS)	agreed

CH	4.3, note , line 1	correct exponent: "…factor of 10 to 10^3 lower …"	We believe there is a font problem between us and we have modified the document to say ten to a thousand times lower for clarity.
CH	4.3, title	add accepted abbreviation: Graphite furnace AAS system (GF-AAS)	Abbreviation added.
CH	4.4, title	add accepted abbreviation: Hydride generation AAS system (HG-AAS)	Abbreviation added.
CH	4.5, title	add accepted abbreviation: Cold vapor AAS system (CV-AAS)	Abbreviation added.
AU	5.6	Is the "blank" referred to the 'blank reference solution' or 'blank test solution'?	blank test solution, text has been modified.
CH	5.7, line 7,8	use italics for correlation coefficient: r correct exponent: r^2	Our document has italics and the correct exponent. Once again we may have a font problem.
CH	Table I	add remark: For certain elements such as sodium or lithium an ionisation suppressor such as cesium may be necessary in order to increase sensitivity and linearity.	Footnote has been added
UK	Table II	Comment a, Line 3, should say "for example a sample of 10 µL" not litres.	Font disparity again?
CH	6.1.1	add remark: For safety reasons cylinders of compressed acetylene (ethyne) are always shipped and stored in which the acetylene is dissolved in acetone or dimethylformide (DMF).	Note has been added.

CH	6.5	An additional requirement should be added: The raw data, that is the data before any internal data manipulation shall be accessible for the user in order to facilitate data treatment by users. If raw data are internally transformed the type of software treatment has to be described so that data transformations are fully transparent. Data transformations based on statistical procedures and corrections for systematic influences are to be outlined for the user.	It is too late in the process to add additional requirements, but the Secretariat shall take it under advisement to add this in future revisions.
UK	6.7	Comment including input from LGC as a current user of AAS Instrumentation. We still believe that a minimum period of availability should be agreed for manuals and parts. This Recommendation is likely to impact on public analysts employed by local authorities, and on their counterparts in SMEs, as well as laboratories in developing countries who often have no regular capital budget. They may be forced to depend on a single spectrometer for 20 years or more. A long-term commitment to support these instruments could help manufacturers meet environmental and customer care goals.	See comment to 6.5. We are sympathetic to your concern but OIML is focused on metrological requirements and manufacturers must respond to complex economic forces where different components may become obsolete without notice. Such a requirement as you propose would exceed the scope of OIML.
CH	References	add a title for a classic book on the subject: Jiří Dědina, Dimiter L. Tsalev, Hydride generation atomic absorption spectrometry, John Wiley &Sons, (1995), ISBN 0471-95364-4	Agreed
CH	A.1.1	Add one more remark: The stock solutions have to be compared with suitable certified reference materials in order to establish traceability of the calibration material. Stock solution with known certified values are preferred.	Agreed
AU	B.2	Should B.2.3, B.2.4 and B.2.5 instruct the user to zero the instrument? The sections do instruct the user to set up the instrument according to B.2.2.1. However B.2.2.1 doesn't instruct the user to zero the instrument (this instruction is located at B.2.2.2).	Zero instructions have been moved from B.3.2.2 to B.3.2.1.

AU	B.2.1	Should "(c) blank reference solution of 0.1%" be '(c) blank test solution of 0.1%'?	B.3.1 blank test solution
AU	B.4.1	Should "(c) blank reference solution of 0.1%" be '(c) blank test solution of 0.1%'?	B.3.1 blank test solution
CH	B.2.3.1	As we do not deal with the population but with a limited random sample the symbol used has to be a "*s*" and not the greek "σ": Small letters instead of capital letters are used (cf. ISO-GUM). $s_r = \frac{s}{\bar{x}} \cdot 100\%$ where $s = \left[\frac{\sum (x_i - \bar{x})^2}{n-1}\right]^{1/2}$ and $\bar{x} = \frac{\sum x_i}{n}$ Accordingly all symbols used in the text and formulas of chapter B2.5 and the rest of the document have to be adapted.	Agreed
CH	B.2.5	A helpful parameter is the residual standard deviation s_0: $s_0 = \left[\frac{\sum (y_i - \hat{y}_i)^2}{n-2}\right]^{1/2}$ in which the expectation value $\hat{y}_i$ is the value given by the linear regression line. With this parameter and the slope S of the linear regression line the practical detection limit DL can be given. This detection limit is always larger than the one based on the noise of the base line as declared in 3.6. $DL = 3 \cdot \frac{s_0}{S}$ To evaluate this detection limit only the calibration solutions are need and no special blank solution. This practical detection limit has to be reported in annex C in addition to the one deduced from the instrument noise measurement.	It is too late in the process to add additional requirements, but the Secretariat likes the suggestion of the addition of the residual standard deviation and will take it under advisement to add this in future revisions.

UK	B.4.4.1 line 2	Further Comment including input from LGC as a current user of AAS Instrumentation. Some operators calibrate the instrument first, and obtain these data in concentration units. Therefore delete ‘absorbance’. Likewise at B.4.4.1. A linked amendment is needed to the next paragraph (B.4.4.1.) to say ‘by 3 and, if it was initially expressed in absorbance units, by’. We still believe that these amendments are necessary.	Agreed.