BS EN 15297:2011

BSI Standards Publication

# Solid biofuels — Determination of minor elements — As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V and Zn

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This British Standard is the UK implementation of EN 15297:2011. It supersedes DD CEN/TS 15297:2006 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/17, Solid biofuels.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 71235 7

ICS 75.160.10

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 28 February 2011.

**Amendments issued since publication**

| Date | Text affected |
|---|---|

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

# EN 15297

February 2011

ICS 75.160.10

Supersedes CEN/TS 15297:2006

English Version

# Solid biofuels - Determination of minor elements - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V and Zn

Biocombustibles solides - Détermination des éléments mineurs - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V et Zn

Feste Biobrennstoffe - Bestimmung von Spurenelementen - As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, V und Zn

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EUROPÄISCHES KOMITEE FÜR NORMUNG

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# Contents

Page

# Foreword

This document (EN 15297:2011) has been prepared by Technical Committee CEN/TC 335 “Solid biofuels”, the secretariat of which is held by SIS.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2011, and conflicting national standards shall be withdrawn at the latest by August 2011.

This document supersedes CEN/TS 15297:2006.

In the pre-normative project BIONORM I&II a robustness test has been performed to find out if all critical parameters in the standard were addressed. Based on the results of that test it has been concluded that all critical parameters were covered. Only minor technical changes were necessary which have been implemented in the revised text. The revision also includes a change of deliverable from Technical Specification to European Standard and updated normative references.

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# Introduction

The minor elements present in solid biofuels can in some cases be of environmental concern, e.g. it has been shown that certain energy crops will concentrate cadmium and in polluted areas other toxic elements may be found at elevated concentrations in the biofuels. This can be a problem if, for example, the ash from the combustion is to be put back in the forest as a fertilizer. Trace elements in biofuels are often present at very low concentrations requiring great care to avoid contamination in the sample preparation and decomposition steps. The typical concentrations of minor elements in solid biofuels can be found in EN 14961-1 [1]. In this European Standard wet chemical methods are described. Alternative methods such as X-ray fluorescence (XRF) or direct mercury analysers may be used when validated with suitable materials (biomass reference materials).




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## 1 Scope

This European Standard is intended for determination of the minor elements Arsenic, Cadmium, Cobalt, Chromium, Copper, Mercury, Manganese, Molybdenum, Nickel, Lead, Antimony, Vanadium and Zinc in all solid biofuels. Further it specifies methods for sample decomposition and suggests suitable instrumental methods for the determination of the elements of interest in the digests. The determination of other elements as Selenium, Tin and Thallium is also possible with the method described in this European Standard.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12338, *Water quality — Determination of mercury — Enrichment methods by amalgamation*

EN 14588:2010, *Solid biofuels — Terminology, definitions and descriptions*

EN 14774-3, *Solid biofuels — Determination of moisture content — Oven dry method — Part 3: Moisture in general analysis sample*

FprEN 14780, *Solid biofuels — Methods for sample preparation*

EN 15296, *Solid biofuels — Conversion of analytical results from one basis to another*

EN ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885:2007)*

EN ISO 11969, *Water quality — Determination of arsenic — Atomic absorption spectrometric method (hydride technique) (ISO 11969:1996)*

EN ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements (ISO 17294-2:2003)*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 14588:2010 and the following apply.

**3.1**
**Reference Material**
**RM**
material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials

**3.2**
**Certified Reference Material**
**CRM**
reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence

**3.3**
**NIST Standard Reference Material®**
**SRM**
CRM issued by NIST that also meets additional NIST-specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterisations and provides information regarding the appropriate use(s) of the material

# 4 Symbols and abbreviations

## 4.1 Symbols

As Arsenic

Cd Cadmium

Co Cobalt

Cr Chromium

Cu Copper

Hg Mercury

Mn Manganese

Mo Molybdenum

Ni Nickel

Pb Lead

Sb Antimony

V Vanadium

Zn Zinc

## 4.2 Abbreviations

| | |
|---|---|
| CV-AAS | Cold vapour atomic absorption spectrometry |
| GF-AAS | Graphite furnace atomic absorption spectrometry |
| HG-AAS | Hydride generation atomic absorption spectrometry |
| ICP-OES | Inductively coupled plasma optical emission spectrometry |
| ICP-MS | Inductively coupled plasma mass spectrometry |

# 5 Principle

The analysis sample is digested in a closed vessel made from a fluoropolymer using nitric acid, hydrogen peroxide and hydrofluoric acid in a microwave oven or a resistance oven or heating block. The digest is then diluted and the elements determined with suitable instruments.




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# 6 Reagents

## 6.1 General

All reagents shall be of analytical grade or better. If the blank level is unacceptably high i.e. more then 30 % of the determined value, the use of ultra pure reagents should be investigated.

## 6.2 Water

Water containing negligible amounts of the minor elements i.e. amounts that do not contribute significantly to the determinations. Deionised water or doubly distilled water will normally fulfil this requirement.

NOTE The water used for analytical trace metal work is normally produced using a system for production of ultra pure water for laboratory use conductivity = 0,056 μS/cm.

## 6.3 Hydrofluoric acid (HF)

40 % (w/w), $\rho$ = 1,13 g/ml

**CAUTION — Hydrofluoric acid may lead to health hazards.**

## 6.4 Hydrogen peroxide ($H_2O_2$)

30 % (w/w), $\rho$ = 1,11 g/ml

## 6.5 Nitric acid ($HNO_3$)

≥ 65 % (w/w), $\rho$ = 1,41 g/ml

## 6.6 Boric acid ($H_3BO_3$)

4 % (w/w)

## 6.7 Use of Certified Reference Materials (CRM or SRM)

Use certified reference materials, issued by an internationally recognized authority, to check if the accuracy of the calibration meets the required performance characteristics. Examples of certified reference materials are: NBS 1570 spinach leaves, NBS 1571 orchard leaves, NBS 1573 tomato leaves and NBS 1575 pine needles.

When, due to matrix effects or concentration range limitations, no good recoveries for the certified reference materials can be obtained, calibration with at least two CRM or SRM materials may solve these problems. In that case CRM or SRM materials other than used for the calibration shall be used for verification purposes.

NOTE A CRM or SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

# 7 Apparatus

### 7.1 Heating oven or heating block suitable for the decomposition system in use.

A resistance heated oven or heating block that can be used at a temperature of at least 220 °C and an accuracy of ± 10 °C.




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**7.2 Microwave oven.**

Intended for laboratory use and preferably equipped with temperature control.

**7.3 Sample digestion vessels.**

Intended for the heating system used, normally made of a fluoro plastic.

**7.4 Balance.**

With a resolution of at least 1 mg.

**7.5 Plastic volumetric flasks.**

## 8 Preparation of the test sample

The test sample is the general analysis test sample with a nominal top size of 1 mm or less, prepared in accordance with FprEN 14780. For the milling of the sample special attention shall be taken to the risk of contamination from the inner materials of the mill. These materials shall be chosen depending on the elements to be determined. If for example chromium and nickel have to be determined with a high accuracy at low levels stainless steel materials should be avoided for the parts of the mill having contact with the sample, using for example tungsten carbide or titanium instead. Due to the higher abrasion rate the use of high-speed mills should in general be avoided.

The results are to be calculated on a dry basis. Therefore the moisture content of the test sample shall be determined as described in EN 14774-3.

## 9 Procedure

### 9.1 Digestion

a) Weigh, in the digestion vessel, 400 mg to 500 mg homogenised sample, to the nearest 1 mg.

b) Add 2,5 ml hydrogen peroxide (30 %) and wait 1 min to 5 min.

c) Add 5 ml nitric acid (65 %).

d) Add 0,4 ml hydrofluoric acid (40 %) and close the sample digestion vessel. The hydrofluoric acid may be omitted provided that it can be shown that equivalent results can be obtained for the actual type of solid biofuel. When hydrofluoric acid is used the instrument used for the analysis shall be equipped with components resistant to this.

NOTE 1 For this relatively low concentration of hydrofluoric acid the only modification normally necessary when using ICP-OES or ICP-MS instruments is to use a nebulizer resistant to hydrofluoric acid. The instrument manufacturer can give information regarding the use of hydrofluoric acid.

NOTE 2 It is in some cases possible to use boric acid to complex the hydrofluoric acid, especially when using GFAAS or ICP-OES.

This shall be validated for the actual instrument. Care shall be taken to use boric acid with the necessary purity.

e) Heat the sample according to the following heating programmes for digestion:




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1) Resistance heating[1)]: Step 1: Over 1 h heat to 220 °C, rate 3,33 °C/min

Step 2: Hold for 1 h at 220 °C

NOTE 3 Some available digestion bomb systems use fluoropolymer vessels, which cannot withstand temperatures above 170 °C. In such cases this lower temperature can be used provided it can be shown that comparable results are obtained as when 220 °C is used e.g. by the use of equivalent biomass reference materials.

2) Microwave heating[2)]: Step 1: Over 15 min heat to 190 °C

Step 2: Hold for 20 min at 190 °C

f) After cooling, transfer the digest to a volumetric flask. Rinse the digestion vessel carefully with high purity water and transfer the rinse solution to the volumetric flask. Add high purity water to the digest to an appropriate volume, depending on the detection method to be used.

### 9.2 Detection methods

— As, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se, Sn, Tl, V and Zn can be detected by ICP-MS, ICP-OES or GF-AAS provided that the detection limits of the used method are sufficient for the fuel specifications to be verified.

— As and Se can be determined using HG-AAS according to the principles described in EN ISO 11969.

— Hg can be determined using CV-AAS according to the principles described in EN 12338.

— ICP-OES can be used according to the principles described in EN ISO 11885.

— ICP-MS can be used according to the principles described in EN ISO 17294-2.

— Other instrumental methods may be used after validation with biomass reference material of a suitable type.

### 9.3 Calibration of the apparatus

When the analytical system is evaluated for the first time, establish a calibration function for the measurement in accordance with the manufacturers' instructions. Adjust the established calibration function during the analysis if necessary. Check the performance of the instrument using the accepted standard procedures like replicate analysis, use of SRM and or CRM, control samples and control charts. The calibration and quality control scheme shall be organized and maintained in such a way that the required uncertainty of measurement can be obtained. The results of the validation study of BioNorm2 (Annex A) demonstrates what is achievable with commercial instruments that are used by experienced laboratories.

### 9.4 Analysis of the digests

Analyse the digests in accordance with the manufacturer's instructions.

### 9.5 Blank test

Carry out a blank test, using the same procedure and methods as described in 9.1 to 9.4, but omitting the test portion. This assesses both the contents of the elements in the reagents and any contamination from equipment and in the laboratory atmosphere. The obtained blank value should be subtracted from the value in

1) The stated temperature refers to heating device (e.g. oven).

2) The stated temperature refers to digest solution.

the sample. This contribution shall not be quantitatively significant, if it is more than 30 % of the result the blank value should be reported and the use of higher quality reagents should be considered.

## 10 Calculations

The content of an element in the sample on dry basis, $w_i$, expressed in mg/kg, is calculated from the mean of duplicate determinations using Equation (1):

$$w_i = \frac{(c_i - c_{i,0}) \times V}{m} \times \frac{100}{(100 - M_{ad})} \quad (1)$$

where

$w_i$ is the concentration of the element in the sample, on a dry basis, in mg/kg;

$c_i$ is the concentration of the element, in the diluted sample digest, in mg/l;

$c_{i,0}$ is the concentration of the element, in the solution of the blank experiment, in mg/l;

$V$ is the volume of the diluted sample digest solution, in ml;

$m$ is the mass of the test portion used, in g;

$M_{ad}$ is the moisture content in the analysis test sample in % m/m;

The results may be calculated to other bases e.g. to as received basis according to EN 15296.

## 11 Performance characteristics

The achievable performance of the method is given in Annex A showing the results obtained by a European intercomparison study carried out for a sample of wood chips and a sample of an exhausted olive residue. These two samples represent the extremity of the method. The wood chip sample represents samples with low contents of most of the elements and the olive residue samples with high amounts of most of the elements.

## 12 Test report

The test report shall include at least the following information:

a) identification of the laboratory performing the test and the date of the test;

b) identification of product (or sample) tested;

c) reference to this European Standard (EN 15297);

d) method for the determination;

e) results of the test including the basis in which they are expressed, as indicated in Clause 10;

f) any unusual features noted during the determination;

g) any operation not included in this European Standard, or regarded as optional.




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# Annex A
(informative)

# Performance data

The round robin was carried out by laboratories in Austria, Belgium, Denmark, Finland, Germany, Ireland, Italy, the Netherlands, Spain, Sweden and the United Kingdom. The variety of instruments and other analytical conditions were used in accordance with the quality parameters specified in the method.

The tests were carried out using two samples, wood chips and exhausted olive residues produced in the EU-project BioNorm in 2008 in accordance with CEN/TS 14780. The sample “wood chips” was made of German coniferous wood chips; the chips were dried and milled to 1 mm by means of cutting mill. The sample “exhausted olive residues” was obtained from olive oil industry in Spain from a typical outdoor storage facility. In the original sample stones and other natural impurities were present. These impurities and stones were removed manually and the sample was prepared from the residues in two steps using a coarse cutting mill equipped with a 10 mm sieve and a laboratory cutting mill equipped with WC cutting tools and a 1 mm sieve.

All data is reported on dry basis.

The performance data according to ISO 5725-2 [2] are presented in Tables A.1 - A.13.

NOTE 1 See Table A.1 for definition of the symbols used in Tables A.1 to A.13.

NOTE 2 A guideline can be found in EN 15296 on how to use these validation parameters.

**Table A.1 — Performance data for Arsenic (As)**

| **Sample** | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 6 | 27 | 10 | 0,036 | 0,023 | 64 | 0,012 | 34 |
| exhausted olive residues | 8 | 38 | 7,3 | 0,60 | 0,057 | 9,5 | 0,036 | 6,0 |

| Definition symbols | |
|---|---|
| $n$ | is the number of laboratories after outlier elimination |
| $l$ | is the number of outlier free individual analytical values |
| $o$ | is the percentage of outlying values from replicate determination |
| $x$ | is the overall mean |
| $s_R$ | is the reproducibility standard deviation |
| $CV_R$ | is the coefficient of the variation of the reproducibility |
| $s_r$ | is the repeatability standard deviation |
| $CV_r$ | is the coefficient of the variation of the repeatability |

**Table A.2 — Performance data for Cadmium (Cd)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 13 | 63 | 3,1 | 0,32 | 0,021 | 6,4 | 0,009 | 2,9 |
| exhausted olive residues | 8 | 40 | 0 | 0,025 | 0,005 7 | 23 | 0,005 0 | 20 |

**Table A.3 — Performance data for Cobalt (Co)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 9 | 43 | 4,4 | 0,34 | 0,033 | 9,7 | 0,010 | 2,9 |
| exhausted olive residues | 11 | 54 | 1,8 | 1,04 | 0,128 | 12 | 0,056 | 5,4 |

**Table A.4 — Performance data for Chromium (Cr)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 9 | 43 | 4,4 | 0,37 | 0,12 | 31 | 0,077 | 21 |
| exhausted olive residues | 15 | 72 | 4 | 14,3 | 3,4 | 24 | 1,08 | 7,6 |

**Table A.5— Performance data for Copper (Cu)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 12 | 57 | 6,6 | 1,29 | 0,16 | 12 | 0,091 | 7,0 |
| exhausted olive residues | 15 | 75 | 0 | 25 | 2,2 | 8,6 | 0,85 | 3,4 |

**Table A.6 — Performance data for Mercury (Hg)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 6 | 27 | 3,6 | 0,007 2 | 0,001 6 | 23 | 0,001 0 | 13 |
| exhausted olive residues | 10 | 44 | 2,2 | 0,012 | 0,004 8 | 40 | 0,002 6 | 22 |




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**Table A.7 — Performance data for Manganese (Mn)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 14 | 69 | 1,4 | 261 | 18 | 6,78 | 3,78 | 1,4 |
| exhausted olive residues | 15 | 73 | 2,7 | 40,2 | 2,5 | 6,30 | 1,46 | 3,6 |

**Table A.8 — Performance data for Molybdenum (Mo)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 5 | 23 | 0 | 0,028 | 0,015 | 52 | 0,012 | 41 |
| exhausted olive residues | 8 | 37 | 7,5 | 0,22 | 0,056 | 25 | 0,018 | 8,2 |

**Table A.9 — Performance data for Nickel (Ni)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 10 | 47 | 0 | 0,60 | 0,103 | 17 | 0,042 | 7,0 |
| exhausted olive residues | 11 | 54 | 1,8 | 12,5 | 0,82 | 6,5 | 0,68 | 5,4 |

**Table A.10— Performance data for Lead (Pb)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 9 | 41 | 10,9 | 0,75 | 0,117 | 16 | 0,072 | 9,6 |
| exhausted olive residues | 13 | 56 | 6,7 | 3,83 | 0,575 | 15 | 0,357 | 9,3 |

**Table A.11 — Performance data for Antimony (Sb)**

| Sample | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 5 | 25 | 0 | 0,013 | 0,004 2 | 31 | 0,001 | 10 |
| exhausted olive residues | 5 | 24 | 4 | 0,094 | 0,014 | 15 | 0,010 | 11 |

**Table A.12— Performance data for Vanadium (V)**

| **Sample** | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 9 | 42 | 4,5 | 0,076 | 0,018 | 23 | 0,009 | 12 |
| exhausted olive residues | 11 | 51 | 7,3 | 4,26 | 0,45 | 11 | 0,21 | 4,9 |

**Table A.13— Performance data for Zinc (Zn)**

| **Sample** | $n$ | $l$ | $o$ | $x$ | $s_R$ | $CV_R$ | $s_r$ | $CV_r$ |
|---|---|---|---|---|---|---|---|---|
| | | | % | mg/kg | mg/kg | % | mg/kg | % |
| wood chips | 15 | 70 | 5,4 | 13,8 | 1,94 | 14 | 0,67 | 4,8 |
| exhausted olive residues | 15 | 71 | 5,3 | 18,2 | 1,97 | 11 | 0,83 | 4,5 |

# Bibliography

[1] EN 14961-1, *Solid biofuels — Fuel specifications and classes — Part 1: General requirements*

[2] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[3] ISO/TS 21748, *Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation*

[4] NIST definitions, http://ts.nist.gov/MeasurementServices/ReferenceMaterials/DEFINITIONS.cfm

[5] NIST Technical note 1297:1994, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, *http://www.nist.gov/physlab/pubs/tn1297/index.cfm*

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