



CERTIFIED REFERENCE MATERIAL BCR[®] – 660

CERTIFICATE OF ANALYSIS

WINE ETHANOL (12 % VOL.)			
	Certified value ⁵⁾	Uncertainty ⁶⁾	Number of accepted sets of data
Site-specific deuterium isotope ratio (D/H)_I determined by ² H-NMR ¹⁾	102.90 x10 ⁻⁶	0.16 x10 ⁻⁶	13
Site-specific deuterium isotope ratio (D/H)_{II} determined by ² H-NMR ¹⁾	131.95 x10 ⁻⁶	0.23 x10 ⁻⁶	13
Relative deuterium isotope ratio R determined by ² H-NMR ¹⁾	2.567	0.005	12
δ¹³C_{VPDB} measured by IRMS ²⁾	-26.72 ‰	0.09 ‰	12
Alcoholic grade (volume fraction of ethanol) t_Q ³⁾	11.96 %	0.06 %	9
(D/H)_w water determined by IRMS ⁴⁾	148.68 x10 ⁻⁶	0.14 x10 ⁻⁶	5
<p>1) As determined by following EC/2676/90</p> <p>2) As determined by following CEN/TC 174 N 108</p> <p>3) As determined by following EEC/2676/90 Annex 3 and EEC/2870/00 Annex I</p> <p>4) As determined by following CEN/TC 174 N 110</p> <p>5) Unweighted mean of the means of accepted sets of data, each set being obtained in a different laboratory. The values are traceable to the Vienna Standard Mean Ocean Water (VSMOW) (deuterium ratios) and, via comparison with NBS-22 (using a reference value of -29.73 ‰), to the Vienna Pee Dee Belemnite (VPDB) (carbon isotope ratios), respectively. The value for alcoholic grade is traceable to the International System of Units (SI).</p> <p>6) The uncertainty is taken as the half-width of the 95 % confidence interval of the mean of the accepted datasets.</p>			

This certificate is valid for one year after purchase.

Sales date:

The whole sample has to be used for distillation.

Brussels, June 2001

Latest revision: January 2017

Signed:

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NOTE

This material has been certified by BCR (Community Bureau of Reference, the former reference materials programme of the European Commission). The certificate has been revised under the responsibility of the JRC.

DESCRIPTION OF THE SAMPLE

The sample consists of approximately 370 mL of an aqueous solution of 12 % vol. neutral ethanol from wine origin.

ANALYTICAL METHOD USED FOR CERTIFICATION

The method used for the determination of site specific deuterium ratios of ethanol from wine is given in EEC/2676/90.

The site specific deuterium ratio of a site *i* is defined as $(D/H)_i = D_i/P_i N_H$ where D_i is the number of molecules with a deuterium atom in site *i*, N_H is the number of fully protonated molecules, and P_i is the stoichiometric number of hydrogens (deuterons and protons) at site *i*.

For the ethanol molecule, $(D/H)_I$ represents the site-specific deuterium ratio at site I (isotopomer CH_2DCH_2OH) and $(D/H)_{II}$ the site specific deuterium ratio at site II (isotopomer CH_3CHDOH). $(D/H)_I$ and $(D/H)_{II}$ are obtained from the corresponding signal intensities in the 2H -NMR spectrum using an internal comparison with a precisely known amount of working standard, *N,N,N,N*-tetramethylurea (TMU). TMU is a BCR CRM whose values for this parameter are traceable to the internationally-accepted scale VSMOW (Vienna-Standard Mean Ocean Water, supplied by the International Atomic Energy Agency in Vienna).

$(D/H)_I$ and $(D/H)_{II}$ are commonly expressed in parts per million (ppm).

R is calculated as $2 \times (D/H)_{II}/(D/H)_I$ determined from the ratio of the heights of the peaks in the 2H -NMR spectrum and is therefore dimensionless.

The method used for the determination of carbon-13/carbon-12 ratios of ethanol from wine is given in CEN/TC 174 N 108.

$\delta^{13}C_{VPDB}$ of the sample is determined directly on the carbon dioxide obtained from the total combustion of the ethanol and measured using an Isotope Ratio Mass Spectrometer. The results are traceable to the international VPDB scale (Vienna-Pee Dee Belemnite supplied by the International Atomic Energy Agency in Vienna) expressed in parts per thousand.

$$\delta^{13}C_{VPDB} \text{ ‰} = [^{13}C/^{12}C_{\text{sample}}/^{13}C/^{12}C_{VPDB} - 1] \times 1000$$

All measurements were calibrated using NBS-22 with a $\delta^{13}C_{VPDB}$ reference value of -29.73 ‰.

Alcohol grade t_Q is the volume fraction of ethanol in this sample, expressed in %. The alcohol grade of the distillates is measured either by the Karl Fischer method as recommended in the official 2H -NMR method (EEC/2670/90 Annex 8) or by electronic densimetry (EEC/2870/00 Annex I).

PARTICIPANTS

Preparation of the material, homogeneity and stability studies

Eurofins Scientific, Nantes (FR)

Certification measurements

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Central Customs Technical Laboratory, Prague (CZ)

Chemisches Untersuchungsamt, Speyer (DE)

CSL Food Science Laboratory, York (GB)

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SAFETY INFORMATION

Normal laboratory precautions apply.

INSTRUCTIONS FOR USE

The sample requires distillation prior to the analysis. Before use it can be stored at room temperature. Only one distillation can be performed using the volume provided, but several measurements can be carried out on the distillate provided that it is kept tightly closed and stored at +4 °C.

Note: *The $\delta^{13}\text{C}_{\text{VPDB}}$ values are based on a value of -29.73 ‰ for NBS-22. This value has meanwhile been re-assessed and is now stated as -30.03 ‰. When using the new value of NBS-22 for calibration, a different value for the $\delta^{13}\text{C}_{\text{VPDB}}$ of BCR-660 will be obtained. It is not possible to convert the certified value of BCR-660 based on the current reference value of NBS-22.*

STORAGE

On receipt, the materials should be stored at room temperature.

However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

LEGAL NOTICE

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NOTE

A technical report on the production of BCR-660 is available on the internet (<https://crm.jrc.ec.europa.eu>). A paper copy can be obtained from the JRC, Directorate F on request.

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