Method OIV-MA-BS-09 : R2009

Type II method

Method for the determination of total dry extract of spirit drinks of viti-vinicultural origin: gravimetric method

(OIV/OENO 379/2009)

1. Scope

This method is suited to the determination of the total dry extract in spirit drinks of viti-vinicultural origin whichcontain less than 15 g/L of dry matter.

2. Normative References

ISO 3696:1987 Water for analytical laboratory use - Specifications and test methods.

3. Definition

The total dry extract or total dry matter includes all matter that is non-volatile under specified physical conditions.

4. Principle

Weighing of the residue left by evaporation of the spirit on a boiling water bath and drying in a drying oven.

5. Apparatus and Equipment

5.1. Flat-bottomed stainless-steel cylindrical capsule, of sufficient dimensions to avoid loss of liquid when

evaporating.

5.2. Boiling water bath.

5.3. 25 ml pipette, class A.

5.4. Drying oven.

5.5. Dessicator.

5.6. Analytical balance accurate to 0.1 mg.

6. Sampling and Samples.

Samples are stored at room temperature prior to analysis.

7. Procedure

7.1. Pipette 25 ml of the spirit drink into a previously-weighed cylindrical capsule (5.1). During the first hour of evaporation the evaporating dish is placed on the lid of a boiling water bath so that the liquid will not boil, as this could lead to losses through splattering. Leave one more hour directly in contact with the steam of the boiling water bath.

7.2. Complete the drying by placing the evaporating dish in a drying oven at 105 °C ± 3 °C for two hours. Allow the evaporating dish to cool in a dessicator and weigh the

evaporating dish and its contents.

8. Calculation

The mass of the residue multiplied by 40 is equal to the dry extract contained in the spirit and it must be expressed in g/l to one decimal place.

9. Method performance characteristics (Precision)

9.1. Statistical results of the interlaboratory test

The following data were obtained from an international method performance study carried out on a variety of spirit drinks to internationally agreed procedures.

Year of interlaboratory test	1997
Number of laboratories	10
Number of samples	4

Samples	A	B	C	D
Number of laboratories retained after eliminating outliers	9	9	8	9
Number of outliers (Laboratories)	1	1	2	-
Number of accepted results	18	18	16	18
Mean value ($\bar{x}$) g/l	9.0	9.1	10.0	11.8
		7.8	9.4	11.1
Repeatability standard deviation (s_r) g/l	0.075	0.441	0.028	0.123

Repeatability relative standard deviation (RSD_r) (%)	0.8	5.2	0.3	1.1
Repeatability limit (r) g/l	0.2	1.2	0.1	0.3
Reproducibility standard deviation (s_R) g/l	0.148	0.451	0.058	0.210
Reproducibility relative standard deviation (RSD_R) (%)	1.6	5.3	0.6	1.8
Reproducibility limit (R) g/l	0.4	1.3	0.2	0.6

Sample types
A Brandy ; blind duplicates
B Rum ; split levels
C Grappa ; split levels
D Aquavit ; split levels

10. Bibliography

1. Commission Regulation (EC) N° 2870/2000 of 19 December 2000 laying down Community reference methods for the analysis of spirits drinks, *OJEC of 29 December 2000, L333/20*
2. P. Brereton, S. Hasnip, A. Bertrand, R. Wittkowski, C. Guillou, Analytical methods for the determination of spirit drinks, Trends in Analytical Chemistry, Vol. 22, No. 1, 19-25, 2003