



# CIRCULAR ED 210-7

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## GUIDELINES FOR ALCOHOL DETERMINATION

### *PROTOCOL FOR THE DETERMINATION OF ALCOHOLIC STRENGTHS FOR EXCISE PURPOSES BY DENSITY IN AIR USING A PYCNOMETER*

#### *INTRODUCTION*

This protocol is intended to indicate the manner in which alcohol determination must be conducted for excise purposes using a pycnometer.

The protocol consists of two parts:

- Part A - Guidance to operators to enable them to use the instrument in a manner acceptable to the Minister of the Canada Customs and Revenue Agency for determining alcoholic strengths of unobscured spirits, distillates from beers, liqueurs, etc.; and
- Part B - Information to be recorded and available for inspection by excise officers.

The licensee is to immediately inform the Minister, through an excise officer, if any procedure in this protocol is found at any time to be contrary to the manufacturer's operating instructions for the instrument. The licensee must also discontinue execution of the protocol pending further instructions from the Minister.

#### *SCOPE*

This protocol is for the determination of strength of spirits in accordance with the *Departmental Alcohol Determination Regulations, 1997*. The procedure has been checked over the density (density in air) range 0.7882 g/cm<sup>3</sup> to 0.9971 g/cm<sup>3</sup>.

#### ***PART A: GUIDANCE TO OPERATORS TO ENABLE THEM TO USE THE INSTRUMENT IN A MANNER ACCEPTABLE TO THE MINISTER OF THE CANADA CUSTOMS AND REVENUE AGENCY FOR DETERMINING ALCOHOLIC STRENGTHS OF UNOBSURED SPIRITS, DISTILLATES FROM BEERS, LIQUEURS, ETC.***

##### ***I. APPARATUS AND REAGENTS***

- 1.1 A balance capable of determining mass with an accuracy of  $\pm 0.0001$  g.
- 1.2 A glass pycnometer with a nominal capacity of 25 or 50 mL, designed to determine the mass of a liquid at  $20^{\circ}\text{C} \pm 0.05^{\circ}\text{C}$ . The pycnometer consists of a bottle with a flat bottom, an elongated neck on the upper part, and a removable tightly fitted stopper on the top of the elongated neck. The pycnometer must have a unique serial number which is legibly and indelibly marked on its main part and on its stopper.

The pycnometer must be an "approved" pycnometer as required under the *Departmental Alcohol Determination Regulations, 1997* before being used for the determination of alcohol strength for excise purposes.

- 1.3 A constant temperature water bath capable of maintaining a temperature of  $20^{\circ}\text{C} \pm 0.05^{\circ}\text{C}$ .
- 1.4 "Freshly boiled double distilled water" being water which has been freshly boiled after having been:
- distilled twice; or
  - demineralised after being distilled once; or
  - demineralised and passed through a filter to remove organic matter and has a resistivity of 17.5 megohms/cm or more.
- 1.5 Demineralised water or distilled water, herein after called "distilled water".

## 2. *CALIBRATION*

- 2.1 Immerse a clean, dry, stoppered pycnometer in the constant temperature bath, held at  $20^{\circ}\text{C} \pm 0.05^{\circ}\text{C}$ , for one minute, dry the surface with a clean lint-free towel and weigh. Repeat until successive values do not differ by more than 0.2 mg, that is, until constant mass is attained. Record the mass of the empty pycnometer with its proper stopper.
- 2.2 Fill the clean, dry pycnometer with freshly boiled double distilled water, stopper and immerse in the constant temperature bath held at  $20^{\circ}\text{C}$  for 30 minutes. Remove from the bath, adjust the volume of the water in the pycnometer, dry the outside with a lint-free towel, re-stopper and weigh to constant mass. Record the mass in air of the pycnometer filled with freshly boiled double distilled water.
- 2.3 Calculate the mass in air of freshly boiled double distilled water contained in the pycnometer by subtracting the mass in air of the empty pycnometer from the mass in air of the full pycnometer.
- 2.4 Repeat steps 2.1 to 2.3 until at least three values agree within 5 mg or better. Calculate the mean of the three determinations and record the mean as the mass in air of freshly boiled double distilled water or the "water value of the pycnometer".
- 2.5 Calculate the volume of the pycnometer by dividing the mean mass in air of freshly boiled double distilled water (water value of the pycnometer) by the density in air of water ( $0.99715 \text{ g/cm}^3$ ).

## 3. *SAMPLE MEASUREMENT*

- 3.1 At the start of each day, obtain the mass in air of freshly boiled double distilled water at  $20^{\circ}\text{C} \pm 0.05^{\circ}\text{C}$  in exactly the same way as the mass in air of freshly boiled double distilled water was obtained in the calibration procedure. Record mass in air. Calculate and record the density in air of freshly boiled double distilled water by dividing the mass by the volume determined in 2.5. The density in air of freshly boiled double distilled water should be  $0.9971 \text{ g/cm}^3 \pm 0.0001 \text{ g/cm}^3$ . If it is outside  $\pm 0.0001$  and no apparent reason for the discrepancy is found recalibrate the pycnometer as described in 2. and measure a new sample of freshly boiled double distilled water.
- 3.2 Once satisfied that the pycnometer values are within the range of densities for freshly boiled double distilled water, obtain the mass in air of samples at  $20^{\circ}\text{C} \pm 0.05^{\circ}\text{C}$  in exactly the same way as the mass in air of freshly boiled double distilled water was obtained in the calibration procedure. Record masses in air.

- 3.3 Repeat the above determination until at least three values agree within 5 mg or better. Calculate the mean of the three determinations and record the mean as the mass in air of the sample. Calculate and record mean densities of samples.

**PART B: INFORMATION TO BE RECORDED AND AVAILABLE FOR INSPECTION BY EXCISE OFFICERS**

- 1 Each day that the instrument is used for alcoholic strength measurements for excise purposes, record the following parameters:
- 1.1 the date;
  - 1.2 the name of the operator;
  - 1.3 the water values of the pycnometer and the calculated density in air of freshly boiled double distilled water, to 4 decimal places; and
  - 1.4 the masses in air and the calculated density in air of the samples measured, to 4 decimal places.