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Getränkeanalytik

Determination of alcohol content in liqueur and sugared spirits

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page 1/2

Technical information and instructions for use

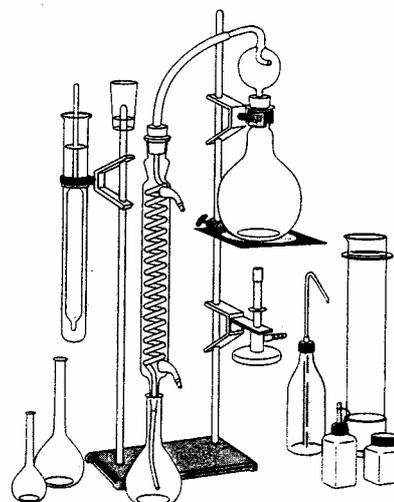
General notes:

The alcohol content in liqueurs cannot be determined directly areometrically by means of alcoholometers ("spindles"). The extract substances contained in these liquids (sugar, proteins, tanning agents, etc.) falsify the measured results. Yet the declaration of the alcohol contents of liqueurs has to be indicated with a tolerance of $\pm 0.3\%$ vol A. The precision required by the legislator can indeed be achieved with a thoroughly executed sample distillation and subsequent areometrical alcohol measurement by using suitable EU alcoholometers.

Because of their viscous consistency and the high sugar content liqueurs represent above all problems with regard to the determination of the alcohol content. Already when measuring the sample volume to be distilled, minor faults can lead to imprecise results. Since the density of alcohol decreases substantially with an increasing temperature, an exact tempering of the sample to 20°C before measuring the volume to be distilled is indispensable. The distillation proves to be difficult when diluting the sample insufficiently, since the liqueur tends to burn at the bottom of the boiling flask. If an insufficient volume is distilled over, the determined alcohol content is too low. The method developed by Schliessmann for the determination of the alcohol contents in liqueurs by a sample distillation takes the above-mentioned particularities of liqueurs into account.

Preparation of the sample distillation equipment:

- Screw aluminium bars onto the plate stand.
- Attach the heating source to the longer aluminium bar (stand heating plate SH 85 or gas burner with ceramic wire netting) and the round clamp with quick opener for the distillation flask.
- Attach the cooler to the shorter aluminium bar by means of the round clamp. The standard ground joint must show upward.
- Connect the cooler with the enclosed hoses and transmission pieces to the water conduit. The water is supplied at the lower connection piece, the water discharge at the upper one.
- Place the distillation attachment with the plug at the hose end onto the cooler.

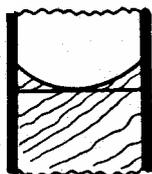


ill.1: Sample distillation equipment

Instructions for the determination of alcohol in liqueurs:

- Temper liqueur sample (approx. 100 ml) in a closed recipient **to exactly 20°C** (approx. 30 minutes in the water bath).
- Fill a 500-ml measuring cylinder up to the 240-ml mark with distilled water. The liquid quantity serves the rinsing-out of the 80 ml measuring flask, in which the sample is measured, without leaving any residues.
- Fill tempered sample if possible **without bubbles** into the 250-ml polyethylene washing bottle and screw-on outlet tip.
- Introduce bottle outlet into the neck of the 80 ml measuring flask until the tip is underneath the ring mark and that during the filling the inner flask wall will not be netted above the ring mark.
- Fill the measuring flask with the sample liquid by slightly pressing the PE washing bottle. **Avoid formation of bubbles!**
- Set meniscus exactly onto the ring mark (compare ill. 2).

Reading



„Meniscus on mark“

ill. 2: Reading of the meniscus

- Pour measured liqueur sample into the distillation flask.
- For rinsing, fill measuring flask with water from the 500 ml measuring cylinder, shake the rinsing liquid and transfer it into the distillation flask. Repeat procedure until the distilled water has been fully transferred into the distillation flask.
- To avoid burning and forming of foam during distillation, add 8-10 small boiling stones of type A and 2-3 drops of silicone antifoam solution into the distillation flask.
- Fill approx. 5 ml of distilled water into the receiver flask (200 ml measuring flask) and place the flask in such a way underneath the cooler that the silicone hose at the outlet plug of the cooler reaches into the neck of the measuring flask and just touches the water surface.
- To avoid alcohol losses by evaporation it is recommendable to place the receiver flask into a container filled with an ice/water mixture. The receiver flask can be weighted with lead rings to prevent it from tumbling down.

- Connect distillation flask to the distillation attachment. Make sure that the connection is absolutely tight.
- Switch-on water-cooling.
- Switch-on heating source (stand heating plate SH 85 to level 12).
- As soon as the receiver flask is half filled, it has to be placed in such a low position that the silicone hose at the cooler end reaches in the flask only up to the ring mark and the distillate drops freely.
- Remove receiver flask as soon as it is filled up to approx. 2 cm underneath the ring mark (duration approx. 45-60 minutes).
- Switch-off heating source.
- Close receiver flask tightly with a rubber plug and temper it to 20°C (at least 30 minutes in the water bath).
- Refill with distilled water exactly up to the ring mark.
- Close flask again, shake it well and transfer liquid completely into the dry spindle cylinder 360 x 36 mm.
- Determine alcohol content with alcoholometer of EU class III according to DIN 12803. Read temperature of the sample and implement, if necessary, a temperature correction by means of the official alcohol tables.
- The alcohol content of the liqueur is obtained by multiplying the measured value of the distillate (% vol A) at 20°C with factor 2.5:

e.g. measured value 10% vol A x 2.5 = 25% vol A

Since the alcoholometer of EU class III only covers a measuring range of 5% vol, three alcoholometers are required for measuring liqueurs and extract-containing spirits. The following table shows the measuring range and the required alcoholometers.

Alcohol content of liqueur	Required alcoholometer EU class III
14,0 - 25,0 % vol A	5 - 10 % vol A
25,0 - 37,5 % vol A	10 - 15 % vol A
37,5 - 50,0 % vol A	15 - 20 % vol A

Halving all volumes will reduce the distillation time to the half. Thus 100 ml of distillate is obtained from 40 ml sample and 125 ml water. The same is tested by means of an alcoholometer for 100 ml of distillate in a spindle cylinder 250 mm x 30 mm.

For the measurement of 40 ml of the sample you need a 40 ml- instead of a 80 ml measuring flask and for the collection of the destillate you need a 100 ml- instead of a 200 ml measuring flask (receiver flask).