Distillation of red wine

Aims of the experiment

- To get to know a thermal separation method
- To produce spirits from red wine by distillation
- To observe and understand a distillation
- To determine the boiling point of a mixture of substances

Principles

Distillates from red wine are among the oldest spirits in the world. Even as long ago as 1000 years AD, wine was distilled for the production of spirits in order to achieve a higher ethanol content.

Distillation is a thermal separation method that makes use of the fact that two liquids can have different boiling points. In order to understand this, the boiling point of a liquid must first be defined.

To determine the boiling point of a liquid, the liquid under investigation is heated. The liquid initially evaporates at the surface. The liquid changes from the liquid phase into the gas phase. When after some time the vapour pressure of the liquid is equal to the surrounding pressure, the whole of the liquid can evaporate. The liquid begins to boil. When this situation occurs, the boiling point of the liquid is reached. The boiling point temperature is measured in the rising vapour. This is important, as the vapour at this point is at a temperature which just prevents it from condensing. It is in equilibrium with the boiling liquid.

The liquid in this experiment is red wine, which mainly consists of two components; water and ethanol. The boiling point of pure water at standard atmospheric pressure is 100 °C, and of pure ethanol 78.4 °C. Because of this difference in boiling points, it is possible to separate water and ethanol through distillation.

If red wine is heated to its boiling point, an ethanol-water mixture initially evaporates which consists of 96% ethanol and 4% water. The boiling point of this mixture is 78.2 °C. It is not possible to distil off pure ethanol, as ethanol and water form an azeotropic mixture. The liquids have an azeotropic

Fig. 1: Experimental apparatus for the distillation of red wine.

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proportion ratio at which they can no longer be separated by distillation.

At the start of the distillation, a large amount of alcohol is distilled off with a small amount of water. The result of this is that the proportion of water in the liquid continually increases and the composition of the heated red wine changes. This then causes a change in composition of the vapour. The ethanol content falls and an increasing amount of water is distilled off. This is reflected in the increasing boiling point, as this then approaches the boiling point of water. The spirit will therefore be diluted during the course of the distillation.

Risk assessment

The chemicals used are in general non-hazardous. However, the concentrated ethanol could be flammable.

Equipment and chemicals

1	Pocket-CASSY 2 Bluetooth	524 018
1	CASSY Lab 2	524 220
1	NiCr-Ni adapter S, type K	524 0673
1	Temperature probe, NiCr-Ni 1.5 mm	529 676
1	Claisen distillation bridge 250 mm	665 338
1	Round-bottom flask, 250 mL, NS 19/26	664 301
1	Round-bottom flask, 100 mL, NS 19/26	664 300
1	Joint clip, plastic, NS 19/26 from set	665 391ET10
2	PVC tubing 7 mm diam., 1 m	604 501
2	Hose clamp 12 mm	604 460
1	Screw cap, GL 18, mB	667 305
1	Silicone gasket, GL 18/8, set of 10	667 295
1	Protective sleeves for temp. sensors	666 194
1	Heating mantle 250 mL,	666 6522
1	Laboratory stand II	300 76
2	Adhesive magnetic board 500 mm	666 4659
2	Magnetic holder, Size 2, 11…14 mm	666 4662
2	Magnetic holder, Size 3, 18…22 mm	666 4663
1	Panel frame C50, two-level, for CPS	666 425
1	Measuring cylinder Boro 3.3, 250 mL	602 954
1	Boiling stones 100 g	661 091
1	Stopcock grease, 60 g	661 082
1	Evaporation dish, porcelain, 72 mL	608 311
1	Wooden sticks, 100 pcs.	672 2520
also required:		
	Red wine	
	Igniter	
1	PC with Windows XP, Vista, 7 or 8	
Also necessary for wireless measurement:		
1	Battery for Pocket-CASSY 2 Bluetooth	524 019
1	Bluetooth dongle	524 0031

Set-up and preparation of the experiment

Construction of the apparatus

The distillation apparatus consisting of a Claisen bridge, a 250 mL flask, a 100 mL flask, a temperature probe (with CASSY) with protective sleeves, a laboratory stand and a heating mantle is set up on an adhesive magnetic board (see Fig. 1). Grease the ground-glass joints between the bridge and the flasks with stopcock grease and attach joint clips. Connect the Claisen bridge in reverse flow to a cold water tap. Connect the temperature probe via the Pocket-CASSY to the PC.

Performing the experiment

1. Load CASSY Lab settings.

2. Remove the 250 mL flask, add 200 mL of red wine using the measuring cylinder and five boiling stones. Then locate the heating mantle under the filled flask by raising it on the jack-stand.

3. Start the measurement. Open the water tap slightly to perform the distillation with a slow reverse flow. Finally switch on the heating mantle at its highest setting.

4. When a sufficient quantity of spirits has distilled off and collected in the 100 mL flask, the measurement can be stopped.

Note: It is advisable rather to terminate the distillation a little earlier, as otherwise the flammability test might not succeed owing to there being too much water in the distillate.

5. Pour some of the distillate into the porcelain dish and test its flammability by igniting it with a burning wooden stick.

Observation

By heating the red wine, the liquid changes from the liquid phase to the gas phase during the distillation. The physical condition of the red wine therefore changes from liquid to vapour. When the hot vapour meets the cold glass of the flask, it condenses and small droplets of liquid form. The longer the liquid is heated, the more vapour is formed and condenses on the glass of the flask. The liquid droplets increase in size and run back into the red wine. With time, the hot vapour rises even higher, as the glass of the flask and the vapour become hotter. After a while, the vapour condenses on the protective glass sleeve and heats up the temperature probe. This increase in temperature is recorded. It corresponds with the boiling point of the red wine. The vapour then flows into the Claisen bridge where it condenses owing to the cooling water and flows into the collection flask. In contrast to the red wine, the condensate is clear.

Evaluation

The evaluation is performed in CASSY Lab. A diagram has been prepared for this.

Determination of the boiling point of the vapour

Firstly set a vertical mark in the "Boiling Point" diagram (right click in the diagram to open the context menu, + → Set Marker, − → Horizontal Line) to determine the boiling point of the liquid (see Fig. 2).

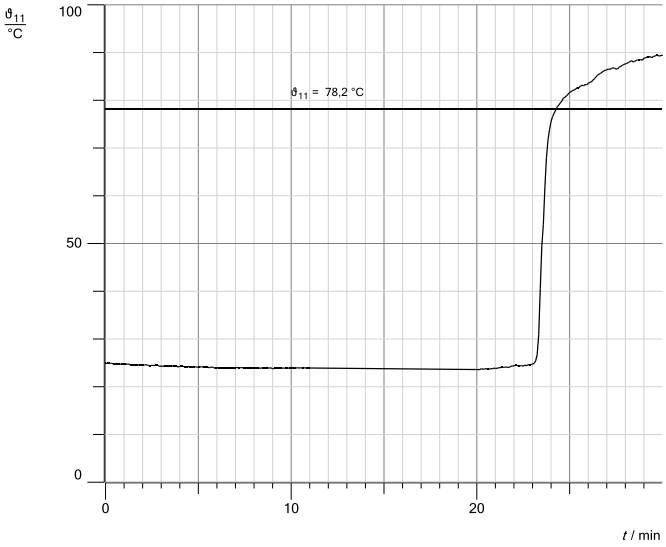

Fig. 2: Recording the temperature against time for the distillation of red wine. Horizontal line: approximate boiling point of Ethanol.

Here we are dealing initially with the vapour from an ethanol-water mixture that is composed of 96% ethanol and 4% water. It boils at about 78 °C. As a large amount of ethanol is initially distilled off with only a small amount of water, an increasing proportion of water remains in the red wine. The composition of the wine changes, with the result that the composition of the vapour also changes. The proportion of water that is distilled off increases, which results in an increase in the boiling point. The distillate therefore becomes more diluted in the course of time.

Following the distillation, a flammability test is performed to establish whether the distillate contains more than 40% ethanol.

Results

In the distillation of red wine, it is not possible to distil off pure ethanol, but only an ethanol-water mixture. This has the initial composition of about 96% ethanol and 4% water. The boiling point of this mixture is 78.2 °C. During the distillation it is also possible to observe that the boiling point of the mixture rises, from which fact a change in the composition of the liquid red wine and of the vapour can be inferred. Following this, the evidence of flammability shows that the distilled spirit contains more than 40% ethanol.

Cleaning and disposal

The wine and the spirits can be disposed of in the laboratory drain.