

KV 1.04

Materials			Support clamp for small casings	02043.10	1
Frame for complete experiments	45500.00	1	Support rod, stainless steel 18/8,		
Rear cover for complete experiment panel	45501.00	1	l = 250  mm	02031.00	1
Panel for complete experimental set-ups	45510.00	1	Cobra3 POWER SUPPLY	12151.99	1
Clamping holder, $d = 1825 \text{ mm}$	45520.00	2	Data cable RS 232, Sub-D/USB	07157.01	1
Clamping holder, $d = 1825$ mm, turnable	45521.00	1	Round bottom flask, short necked,		
Apparatus holder, variable	45526.00	1	DURAN, 100 ml, GL 25/12	35841.15	1
Holder for hand-held instruments			Round bottom flask, two-necked		
on fix. magn.	02161.00	1	with side neck angled, DURAN, 250 ml	35843.15	1
Clamp on holder for demonstration board	02164.00	1	Liebig condenser with attachment	35795.15	1
Fixing band, univ. 100 pcs	45535.00	1	Rubber tubing, $d = 6 \text{ mm}$	39282.00	4
Spring plugs, 50 pieces	45530.00	1	Quick-connect hose coupling, $d = 8 \text{ mm}$	47521.00	2
G-clamp	02014.00	2	Hose clip made of stainless steel,		
Immersion probe NiCr-Ni, Teflon	13615.05	2	d = 8-16  mm	40996.01	6
Hand held instrument 2 x NiCr-Ni, RS 232	07140.00	1	Hose clip made of stainless steel,		
Cobra3 DISPLAY UNIT	12154.00	1	d = 12-20  mm	40995.00	1





Heating mantle in metal housing, 250 ml	47550.93	1
Support clamp		
for the housing of heating mantle	47557.01	1
Power regulator	32247.93	1
Funnel, glass, $d = 80 \text{ mm}$	34459.00	1
Boiling stones, 200 g	36937.20	1
Cylinder, BORO 3.3, glass base, 100 ml	36629.00	1
Hydrometer, 0.851.00 g/cm <sup>3</sup>	38254.02	1
Wash bottle, 500 ml	33931.00	1
Water, distilled, 5 I	31246.81	1
Red wine		

# Safety measures







Ethanol is a colourless, easily inflammable liquid with a characteristic pleasant odour, that is miscible in all proportions with water and almost all organic solvents. Vapours can form explosive mixtures with air. Keep away from sources of ignition - do not smoke. Avoid contact with eyes and skin. Wear appropriate protective clothing, protective gloves and protective goggles when working with it.

Observe the detailed information on safety measures in the appendix.

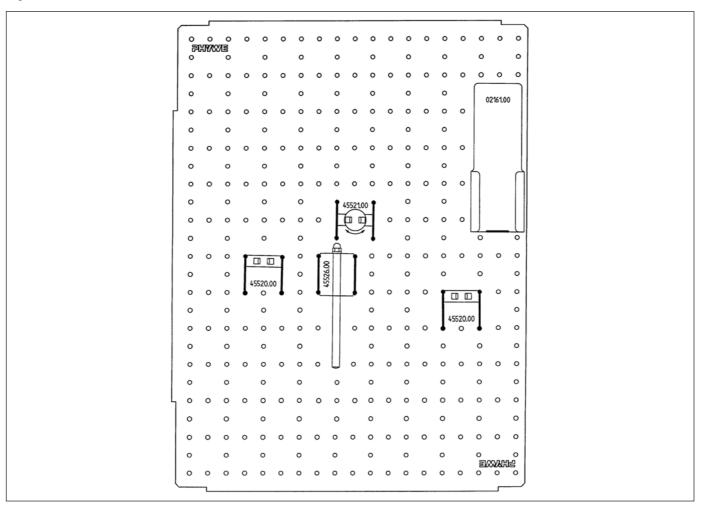
## Set-up

Position the clamping holders on the panel for complete experiments as shown in Fig. 2. The equipment is to be subsequently assembled and fixed to the clamping holders as shown in Fig. 1. Attach the clamp-on holder for the demonstration board to the side of the frame und use the short support rod to hold the Cobra3 DISPLAY UNIT, which has been prepared for this by screwing the support clamp for small casings onto the back of it. Keep as much of the connecting cables as possible behind the panel for complete experiments. Connect hoses for the cooling water to the condenser jacket. Mount two quick-connect hose couplings in this arrangement in order to have the possibility of quick disconnection from the water tap (Fig. 1). Use hose clips to secure all connections against slippage. Fix the tubings to the frame with fixing band.

### **Procedure**

Measure 100 ml of wine into the graduated cylinder and determine the density of the wine with the hydrometer (it may be necessary here to use a hydrometer with a measuring range above 1 g/cm<sup>3</sup>)

Fig. 2





Pour wine into the 250 ml round bottom flask and add about 5 boiling stones. Now set the heating mantle to maximum heating and set the highest step (10) on the power regulator until the first bubbles show that the wine is beginning to boil, then reduce the power to step 8. Carry on with the distillation until a temperature of about 98°C to 100°C is reached in the attachment to the Liebig condenser (this takes approx. 35 minutes).

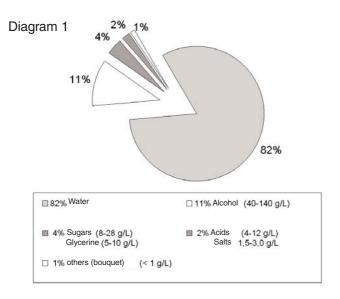
Pour the distillate into the (rinsed) 100 ml graduated cylinder and also wash any remaining distillate into it with distilled water, then make it up to 100 ml with distilled water. Pass the graduated cylinder around the class for an olfactory test.

Determine the density with the hydrometer and read off the alcohol content from the Table.

### Results

Prior to distillation, a wine density of about 1 g/cm<sup>3</sup> is measured

Approx. 20 ml of distillate is collected in the receiver. This smells of alcohol and of other aromatic substances (such as ethereal oils typical for the wine tested). After making it up to 100 ml, a density of 0.980 to 0.990 g/cm<sup>3</sup> is determined. From the Table, this corresponds to an alcohol content of from 10 to 14%.



A practical example could be as follows: A wine has a density of 1.02 g/cm<sup>3</sup> and, according to the label, an alcohol content of 11.5% by volume. After distillation and making up to 100 ml, a density of 0.984 g/cm<sup>3</sup> is determined which, from the Table, corresponds to an alcohol content of 12.2%.

## **Explanation**

When the density of a wine is determined, it corresponds to an alcohol content of approx. 0%. This is because of the composition of wine. The effect of alcohol in the wine is counterbalanced by that of other constituents, such as sugar, acids, ethereal oils etc. (see Diagram 1).

The present regulatory procedure is also based on distillation and a density determination. The wine is first titrated to neutrality with bromphenol blue as indicator, transferred to the distillation apparatus and two thirds of the wine sample then distilled into the receiver. The distillate is then made up to the original volume and the density determined with a pycnometer. The alcohol content is obtained from reference Tables (see Table 1).

#### **Notes**

In this student experiment, accuracy has been sacrificed for simplicity and greater clarity. The following systematic errors have been hereby made:

- Distillation to a boiling point of 100°C.
  - This results not only in alcohol being completely distilled off, but also in other volatile components being carried across into the receiver. Depending on the components, the effect can either be an increase or a decrease in the alcohol content determined. The distillation of two thirds into the receiver (see above) is not recommended because of the time it takes.
- According to the room temperature, alcohol can evaporate from the receiver.
  - Cooling of the receiver should be obligatory. The effect would hardly be noticeable, however, and would lead to a higher density and so to a lower alcohol content.

(Table 1, from: E. Merck, "Useful Tables for Chemical Laboratories")

Density / g⋅cm <sup>-3</sup>	Ethanol, % by vol	Ethanol, % by vol	Density / g⋅cm <sup>-3</sup>	Ethanol, % by vol	Ethanol, % by vol
1.00000	0	0	0.98653	8	10.0
0.99813	1	1.3	0.98505	9	11.2
0.99629	2	2.5	0.98361	10	12.4
0.99451	3	3.8	0.98221	11	13.6
0.99279	4	5.0	0.98084	12	14.8
0.99113	5	6.2	0.97948	13	16.1
0.98955	6	7.5	0.97816	14	17.3
0.98802	7	8.7	0.97687	15	18.5

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## Distillation - Determination of alcohol in wine



- The measuring instruments are only semi-quantitative. A 100 ml graduated cylinder is sufficiently accurate for teaching purposes, but a volumetric flask is more suitable for density determinations (i.e. mass and volume measurements) to three decimal places. The density determination can alternatively be made using a balance and a volumetric flask. In this case, however, care must be taken to minimize transfer losses from one glass vessel to the next!
- The hydrometer scale only allows readings to be made to two decimal places.
  - The third decimal place must be estimated. This is an error that has an appreciable effect, as a deviation of  $0.005~g/cm^3$  can result in a difference of 0.5~to 0.7%

- alcohol. A pycnometer is considerably more accurate, but increases the measuring expenditure.
- The Table used has gaps.
   Values between these must be estimated.

### **Variations**

a) For a minimization of error by an increased separation efficiency:

A column is used to increase the separation efficiency of the distillation apparatus. This requires the following additional materials:

Clamping holder 45520.00 2 Vigreaux-column, l = 200 mm, GL 25/12 35792.15 1

Fig. 3





Positioning of the holders as in Fig. 4. Equipment set-up as in Fig. 3.

Alongside the change in the experimental set-up, a longer distillation time is required with the Vigreux-column, to a total of approx. 45 minutes. This results from the necessity to reach equilibrium in the column. The procedure and evaluation are otherwise exactly as previously.

b) With a distillation in which two or more substances are to be separated:

To separate mixtures containing two or more components, a fractionated distillation having the possibility to collect several fractions of distillate should be carried out. The following are then additionally required:

Clamping holder	45521.00	1
Short condenser, with attachment	35818.15	1
Adapter for 4 flasks, GL 25/12	35869.15	1
Round bottom flask, short-necked,		
DURAN, 50 ml, GL 25/12	35840.15	4

The following are no longer required:
Clamping holder 45520.00 2
Round bottom flask, short necked,
DURAN, 100 ml, GL 25/12 35841.15 1
Liebig condenser with attachment 35795.15 1

Positioning of the holders as in Fig. 6. Equipment set-up as in Fig. 5.

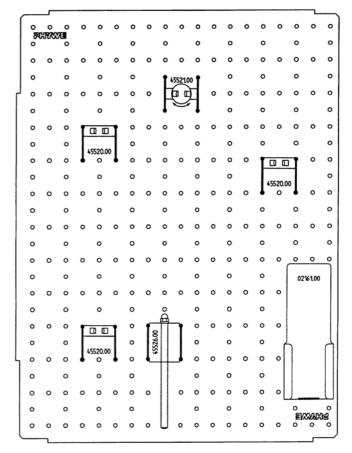
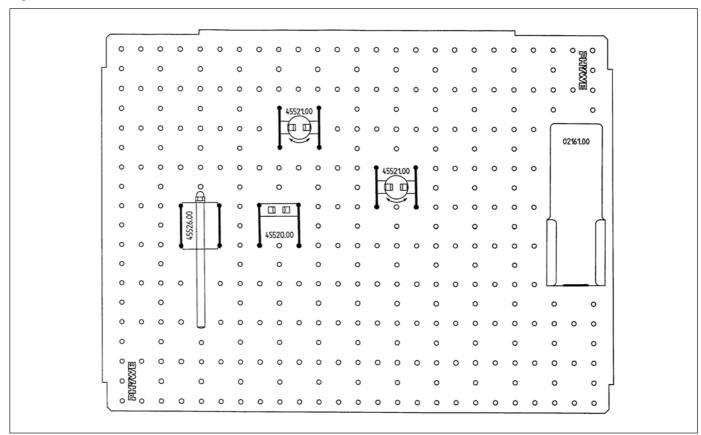


Fig. 4

Fig. 6





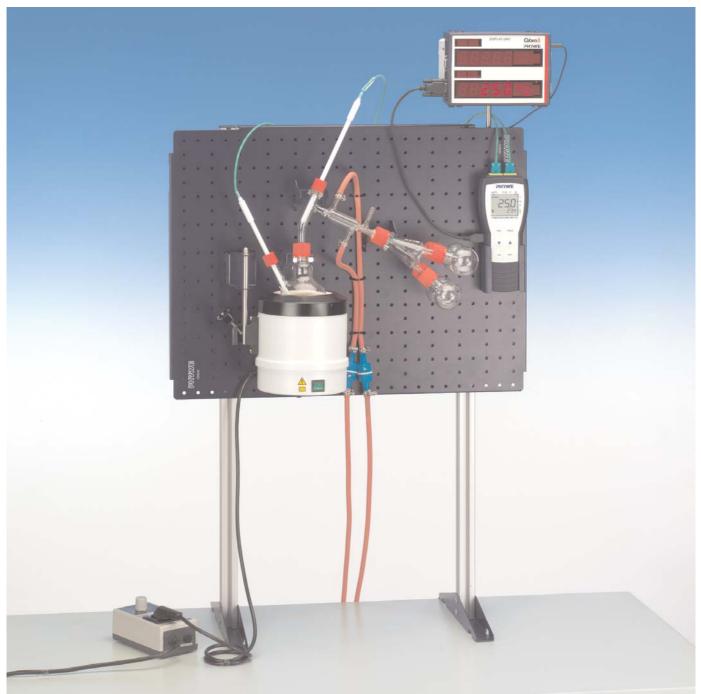


Fig. 5