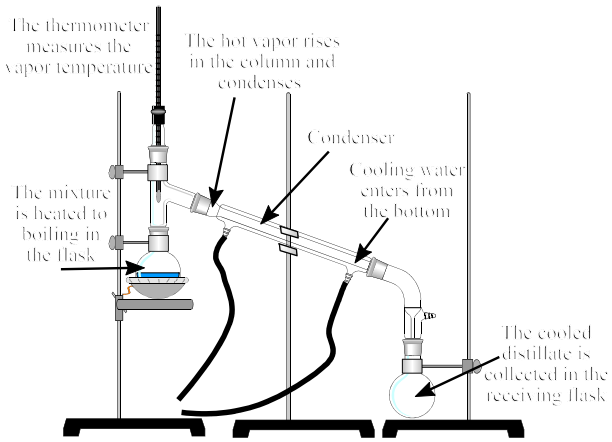
SIMPLE DISTILLATION

It is usually difficult to prepare many organic compounds as pure substances. Few Organic reactions are quantitative. As result, it is necessary to separate one component in the reaction from the others. In this experiment, you will separate the product, ethyl alcohol, from the excess water and unreacted grape juice remaining from the fermentation reaction. This is accomplished by a process known as distillation. In distillation, a liquid is boiled in a closed system. The resulting vapor consists of a mixture of all the volatile materials in the flask. This vapor is then condensed and the distillate collected in a separate container.

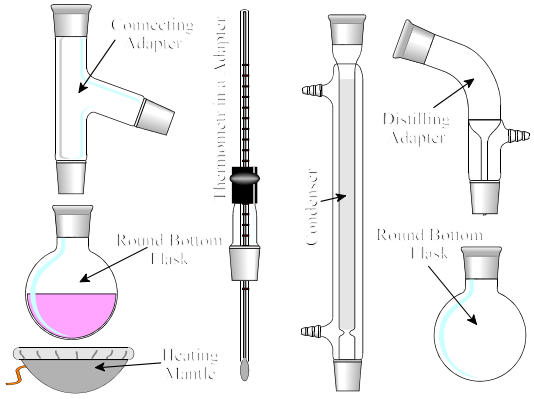


A typical distillation apparatus

The vapor, however, does not have the same composition as the original mixture. The of the vapor is especially rich in the higher vapor pressure components. In your distillation, alcohol has a much higher vapor pressure than the water. Consequently, your distillate has a much higher percentage of alcohol than water. Consult Table #2. Note that is the water-alcohol solution boils at 80°C, the vapor contains 83% alcohol. It the solution boils at 98°C, the vapor contains 19% alcohol while the solution boiled contains only 1% alcohol.

MATERIALS:

Prepared Homogeneous solution, 250 ml round bottom flask, 100ml round bottom flask, heating mantle and controller, boiling chips, thermometer, adapter to connect the flask with the condenser, thermometer and adapter, condenser, distilling tube adapter, 3-ring stands, 3-clamps, a 3" ring, 50 ml beaker, 1 ml pipet, pipet pump, 50-ml graduated cylinder, 250-ml beaker.

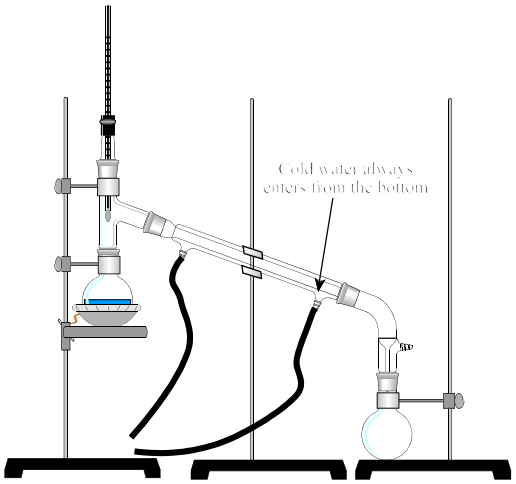


The standard taper glassware needed to assemble the distillation apparatus in this experiment

PROCEDURE:

1. Carefully decant 150 ml of your homogeneous solution into a 250 ml beaker and transfer it into a 250-ml round bottom flask. Add 2-3 boiling chips to the flask to aid the distillation. Measure the mass of 1.00 ml of the your solution product remaining in the soda bottle.

2. Assemble the apparatus as pictured. It is important to lubricate each of the standard taper ground glass joints with a small amount of silicone grease or Teflon tape. To lubricate the joint with silicone grease, place a small dab of lubricant onto the top one-third of one member of the two joining pieces, push the two pieces together and twist to spread the lubricant evenly around the joint. The distillate is collected in a clean, 100 ml round bottom flask.



3. Heat the material at the highest setting of the heating device until the solution appears to be ready to boil. Then lower the heater setting to one-half the original value.

   Note the nature of the droplets of condensate on the sides of the adapter connecting the round bottom flask to the condenser during the progress of the distillation. Continue collecting the distillate, recording the temperature and composition from Table #2 every 30 seconds until you have collected most of the alcohol in your solution. If the temperature drops or no distillate is being collected, increase the heater setting until the temperature rises again or again begins distillate begins to be collected.

   One indication of a change from mostly alcohol to mostly water is the degree of droplet beading on the surface of the distilling column. Water has a surface tension of 72 dynes/cm while pure alcohol has a surface tension of only 28 dynes/cm. The greater the surface tension, the greater the tendency to form droplets. Table #2 also is useful for determining the nature of your distillate.

4. When you have finish the distillation, turn off the heating devise but keep the water running into the condenser until the liquid in the flask is no longer boiling. Pour your distillate into a graduated cylinder and record the total volume of product collected. Mass 1.00 ml of your distillate. Observe both the color and odor of the distillate.

   Carefully remove the 250 ml round bottom flask from the heating mantle and cool it in running water. Observe the color of the spent remaining product before discarding it. What does the color tell you about the effectiveness of the distillation? Carefully transfer the remaining solutions into sample bottles. Label the two containers.

Q1. Calculate both the density of your homogeneous solution before the distillation and the density of your distillate.

Q2. Using Table #3 and the density of your distillate, convert the percent alcohol by mass calculated in question #1 to the percent of alcohol by volume. Determine the "proof" of your distillate. The "proof" equals twice the percent of alcohol **by volume**. A 50% solution would be equal to "100" proof (2 x 50%), etc.

Table 3. SPECIFIC GRAVITY OF ETHANOL-WATER MIXTURES

Specific Gravity (16°C)	Percent Alcohol by Volume	Percent Alcohol by Weight	Specific Gravity (16°C)	Percent Alcohol by Volume	Percent Alcohol by Weight
1.0000	0.0	0.0	0.9344	50.0	42.5
0.9985	1.0	0.8	0.9325	51.0	43.4
0.9970	2.0	1.6	0.9305	52.0	44.4
0.9956	3.0	2.4	0.9285	53.0	45.3
0.9942	4.0	3.2	0.9264	54.0	46.3
0.9928	5.0	4.0	0.9244	55.0	47.2
0.9915	6.0	4.8	0.9222	56.0	48.2
0.9902	7.0	5.6	0.9201	57.0	49.2
0.9890	8.0	6.4	0.9180	58.0	50.2
0.9878	9.0	7.2	0.9158	59.0	51.1
0.9866	10.0	8.0	0.9136	60.0	52.2
0.9855	11.0	8.9	0.9113	61.0	53.2
0.9844	12.0	9.7	0.9091	62.0	54.2
0.9833	13.0	10.5	0.9068	63.0	55.2
0.9822	14.0	11.3	0.9044	64.0	56.2
0.9811	15.0	12.1	0.9021	65.0	57.2
0.9801	16.0	13.0	0.8997	66.0	58.2
0.9791	17.0	13.8	0.8974	67.0	59.3
0.9781	18.0	14.6	0.8949	68.0	60.3
0.9771	19.0	15.4	0.8925	69.0	61.4
0.9761	20.0	16.3	0.8900	70.0	62.5
0.9751	21.0	17.1	0.8876	71.0	63.5
0.9741	22.0	17.9	0.8850	72.0	64.6
0.9730	23.0	18.8	0.8825	73.0	65.7
0.9720	24.0	19.6	0.8799	74.0	66.8
0.9710	25.0	20.4	0.8773	75.0	67.8
0.9699	26.0	21.3	0.8747	76.0	69.0
0.9688	27.0	22.1	0.8721	77.0	70.1
0.9677	28.0	23.0	0.8694	78.0	71.2
0.9666	29.0	23.8	0.8667	79.0	72.4
0.9654	30.0	24.7	0.8639	80.0	73.5
0.9642	31.0	25.5	0.8611	81.0	74.7
0.9630	32.0	26.4	0.8583	82.0	75.8
0.9617	33.0	27.2	0.8554	83.0	77.0
0.9604	34.0	28.1	0.8525	84.0	78.2
0.9591	35.0	29.0	0.8496	85.0	79.4
0.9577	36.0	29.8	0.8465	86.0	80.6
0.9563	37.0	30.7	0.8435	87.0	81.9
0.9549	38.0	31.6	0.8404	88.0	83.1
0.9534	39.0	32.5	0.8372	89.0	84.4
0.9519	40.0	33.4	0.8339	90.0	85.7
0.9503	41.0	34.2	0.8306	91.0	87.0
0.9487	42.0	35.1	0.8272	92.0	88.3
0.9470	43.0	36.0	0.8236	93.0	89.6
0.9454	44.0	36.9	0.8199	94.0	91.0
0.9436	45.0	37.8	0.8161	95.0	92.4
0.9419	46.0	38.8	0.8121	96.0	93.8
0.9401	47.0	39.7	0.8079	97.0	95.3
0.9382	48.0	40.6	0.8035	98.0	96.8
0.9364	49.0	41.5	0.7989	99.0	98.4
0.9344	50.0	42.5	0.7939	100.0	100.0

Table 2. VAPOR-LIQUID COMPOSITION DATA FOR ETHANOL-WATER MIXTURES

Temp °C	% Alc Liquid	% Alc Vapor	Temp °C	% Alc Liquid	% Alc Vapor
78.15	95.57	95.57	86.0	20	72
78.2	91	92	86.5	18	71
78.4	85	89	87.0	17	70
78.6	82	88	87.5	16	69
78.8	80	87	88.0	15	68
79.0	78	86	88.5	13	67
79.2	76	85	89.0	12	65
79.4	74	85	89.5	11	63
79.6	72	84	90.0	10	61
79.8	69	84	90.5	10	59
80.0	67	83	91.0	9	57
80.2	64	83	91.5	8	55
80.4	62	82	92.0	8	53
80.6	59	82	92.5	7	51
80.8	56	81	93.0	6	49
81.0	53	81	93.5	6	46
81.2	50	80	94.0	5	44
81.4	47	80	94.5	5	42
81.6	45	80	95.0	4	39
81.8	43	79	95.5	4	36
82.0	41	79	96.0	3	33
82.5	36	78	96.5	3	30
83.0	33	78	97.0	2	27
83.5	30	77	97.5	2	23
84.0	27	76	98.0	1	19
84.5	25	75	98.5	1	15
85.0	23	74	99.0	0	10
85.5	21	73	99.5	0	5
86.0	20	72	100.0	0	0

NOTE: All percentages are on a weight of alcohol/weight of solution basis

REF: Evans, P.N., J. Ind. & Eng. Chem., **8**, 290-2(1916)