Miscibility gap in a ternary system (Item No.: p3030601)

Curricular Relevance

Area of Expertise: Chemistry
Education Level: University
Topic: Physical Chemistry
Subtopic: Phase Equilibrium
Experiment: Miscibility gap in a ternary system

Difficulty	Preparation Time	Execution Time	Recommended Group Size
Intermediate	10 Minutes	10 Minutes	2 Students

Additional Requirements:

- Precision balance, 620 g / 0.001 g

Experiment Variations:

Keywords:

three component systems, miscibility gap, phase diagram, triangular diagram, Gibb's phase law

Overview

Short description

Principle

A number of completely miscible two component mixtures are prepared to investigate the three component acetic acid / chloroform / water system. These mixtures are titrated with the third component until a two phase system is formed which causes turbidity. The phase diagram for the three component system is plotted in a triangular diagram.

Fig. 1: Experimental set-up.

Safety instructions

Acetic acid, 99…100%
H226: Flammable liquid and vapour
H314: Causes severe skin burns and eye damage
P280: Wear protective gloves/protective clothing/eye protection/face protection.
P308 + P310: If exposed or concerned: Immediately call a POISON CENTER/doctor/…

Chloroform
H315: Causes skin irritation
H319: Causes serious eye irritation
H351: Suspected of causing cancer
P308 + P310: If exposed or concerned: Immediately call a POISON CENTER/doctor/…

Equipment

Position No.	Material	Order No.	Quantity
1	Immersion thermostat Alpha A, 230 V	08493-93	1
2	Bath for thermostat, makrolon	08487-02	1
3	Rack for 20 test tubes, Makrolon	08487-03	1
4	Burette, 10 ml, grad. 0.05 ml	47152-01	2
5	Burette clamp, roller mount., 2 pl.	37720-00	1
6	Retort stand, h = 750 mm	37694-00	1
7	Funnel, glass, top dia. 55 mm	34457-00	1
8	Test tube, 160 x 16 mm, 100 pcs	37656-10	1
9	Rubber stopper,d=18/14mm,w/o hole	39254-00	15
10	Rubber stopper, d=22/17 mm, without hole	39255-00	15
11	Long-neck fl.bott.flask100ml,PN19	36320-00	15
12	Pasteur pipettes, 250 pcs	36590-00	1
13	Rubber caps, 10 pcs	39275-03	2
14	Beaker, high, BORO 3.3, 150 ml	46032-00	1
15	Wash bottle, plastic, 500 ml	33931-00	1
16	Laboratory pen, waterproof, black	38711-00	1
17	Acetic acid 99...100%, pure 1 l	31301-70	1
18	Chloroform 250 ml	48045-25	1
19	Water, distilled 5 l	31246-81	1

Tasks

1. Titrate nine different acetic acid / chloroform mixtures with water until a two phase system is formed in each case.
2. Titrate six acetic acid / water mixtures with chloroform until phase separation is observed.
3. Plot the results of the titrations, expressed as molar fractions, in a trianglular diagram.

Set-up and Procedure

Set up the experiment as shown in Fig. 1.

Prepare the chloroform / acetic acid mixtures listed in Table 1 by weighing the specified quantities of chloroform and pure acetic acid into 100 ml flat-bottomed flasks.

Table 1: Quantities and mixing proportions of chloroform / acetic acid mixtures

Shake the samples well and transfer 10 g of each mixture to suitably labelled test tubes. Temperature equilibrate the test tubes to 25 °C in a temperature controlled water bath. Titrate the samples with distilled water from a microburette until the liquid becomes turbid as a result of separation. Shake the test tubes from time to time to ensure good mixing of the samples (insert rubber stoppers before shaking). Record the quantity of water added (in ml) as approximation for the mass of water (in g).

Repeat the procedure for samples K to P listed in Table 2.

Table 2: Mixing proportions of water / acetic acid

Mixture	$m_{chloroform}/g$	$n_{chloroform}$	$m_{aceticacid}/g$	$n_{aceticacid}$
A	119.5	1.0	0.0	0.0
B	107.6	0.9	6.0	0.1
C	95.6	0.8	12.0	0.2
D	83.7	0.7	18.0	0.3
E	71.7	0.6	24.0	0.4
F	59.8	0.5	30.0	0.5
G	47.8	0.4	36.0	0.6
H	35.9	0.3	42.0	0.7
I	0.0	0.0	60.0	1.0

Again transfer 10 g of each sample to a separate test tube and temperature equilibrate them to 25 °C in the temperature controlled bath. Titrate with chloroform until turbidity occurs and record the quantity added. Calculate the mass of chloroform from the density ($\rho_{chloroform}$ = 1.489 g / ml) and the consumption (in ml).

Calculate the molar fractions for all mixtures and plot the results in a phase diagram using triangular co-ordinates (see Fig. 2).

Mixture	m_{water}/g	n_{water}	$m_{aceticacid}/g$	$n_{aceticacid}$
K	1.00	9.00	10	0.021
L	2.00	8.00	20	0.046
M	3.00	7.00	30	0.076
N	4.00	6.00	40	0.113
O	5.00	5.00	50	0.161
P	6.00	4.00	60	0.22

Theory and evaluation

Gibb's phase law describes the relationship between the number of components K in a system, the phases$P$ formed by the system, and the number of degrees of freedom:

$$P + F = K + 2 \quad (1)$$

where

K Number of components

P Number of phases

F Number of degrees of freedom.

In a three component system ($K = 3$), the sum of phases and the degree of freedom is 5:

$P + F = 5$ (2)

In a single phase system ($P = 1$) under isothermic and isobaric conditions (= 2 degrees of freedom), two degrees of freedom remain; they characterise the composition of the system (molar fractions of two components). If the system has two phases that are under the specified conditions, only one degree of freedom remains for the composition of the system.

The solubility diagram of a ternary system is usually plotted in triangular co-ordinates with equal sides. Each apex of the triangle represents a pure component. The molar fractions of the binary mixtures are entered on the sides of the triangle. The area of the triangle represents all possible mixtures of the system. The following applies for a ternary system:

$x_A + x_B + x_C = 1$ (3)

where

x_i Molar fraction of component i

Both chloroform / acetic acid and water / acetic acid are completely miscible, but chloroform / water are not miscible in any ratio. In the chloroform / acetic acid / water triangular diagram, a straight line is drawn from a point F through the opposite apex K (water), where point F on the chloroform / acetic acid side of the diagram corresponds to a certain composition of the binary chloroform / acetic acid mixture, and point K corresponds to pure water. This straight line represents a constant composition with respect to the components chloroform and acetic acid, whereas the water content rises as one moves along line F – K. To prepare the phase diagram of the chloroform / acetic acid / water system, first enter the molar fractions which correspond to the respective binary mixtures (points A ... P) on the sides of the triangle. Then draw straight lines from these points to the opposing apices. Enter the points which correspond to the molar fraction of the third component and join them. The resulting line separates the two-phase system (below) from the single-phase one (above).

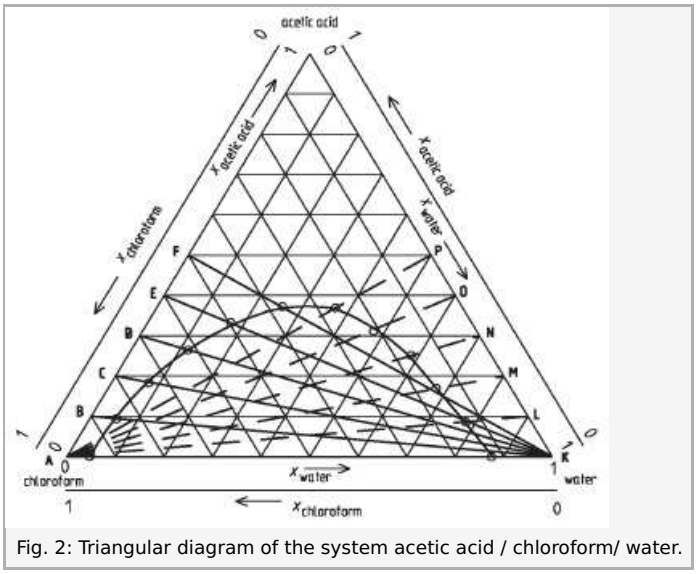

Fig. 2: Triangular diagram of the system acetic acid / chloroform/ water.