





# Miscibility gap in a ternary system



The students learn Gibb's phase law and the miscibility gap in a ternary system.

Physics	Thermodynamics	States of matter, dissolution (kinetic particle theory)	
Chemistry	General Chemistry	Substances mixtures & separation	
Chemistry	General Chemistry	States of matter, dissolution (kinetic particle theory)	
Chemistry	Physical chemistry	Phaseequilibrium	
 Difficulty level medium	 Group size 2	 Preparation time 10 minutes	 Execution time 10 minutes

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## General information

## Application

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Experimental setup

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.

## Other information (1/2)

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### Prior knowledge



The Students should already be familiar with Gibb´s phase law and miscibility in three component systems in theory.

### Scientific principle



A number of completely miscible two component mixtures are prepared to investigate the three component acetic acid / chloroform / water 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.

## Other information (2/2)

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### Learning objective



The students learn Gibb´s phase law and the miscibility gap in a ternary system.

### Tasks



The students titrate nine different acetic acid / chloroform mixtures with water until a two phase system is formed in each case in the first task. Then they titrate six acetic acid / water mixtures with chloroform until phase separation is observed. At last, they plot the results of the titrations, expressed as molar fractions, in a triangular diagram.

## Safety instructions

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- Wear protective gloves/protective clothing/eye protection/face protection.
- For the H- and P-phrases please refer to the corresponding safety data sheets.
- The general instructions for safe experimentation in science education apply to this experiment.

## Theory

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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:

$$P + F = K + 2$$

where

$K$  Number of components

$P$  Number of phases

$F$  Number of degrees of freedom

## Equipment

Position	Material	Item 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 with straight glass stopcock Graduation 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. 50 mm	34457-00	1
8	Test tubes, 160x16mm, 10pcs	36301-03	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	Flat-bottom flask, stopper bed, 250 ml SB 29	MAU-SK17083400	15
12	Pasteur pipettes, 250 pcs	36590-00	1
13	Rubber caps, 10 pcs	39275-03	2
14	Beaker, Borosilicate, tall form, 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	Water, distilled 5 l	31246-81	1

# Setup and procedure

## Setup

Set up the experiment as shown in the figure on the right.

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

Mixture	$m_{\text{chloroform}}/g$	$n_{\text{chloroform}}$	$m_{\text{aceticacid}}/g$	$n_{\text{aceticacid}}$
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	
I	0.0	0.0	60.0	



Click the blue button for a bigger view



Experimental setup

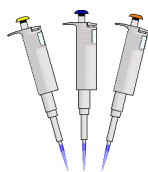
## Procedure (1/2)

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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 the Table on the right.



Mixture	$m_{\text{water}}/g$	$n_{\text{water}}$	$m_{\text{acetic acid}}/g$	$n_{\text{acetic acid}}$
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	0.1
P	6.00	4.00	60	0.22

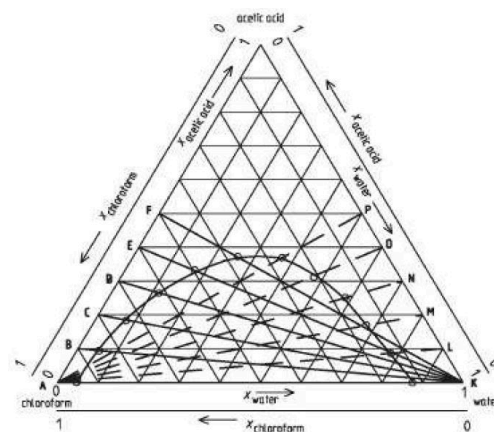
## Procedure (2/2)

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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 ( $P_{\text{chloroform}} = 1.489g/ml$ ) 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. right).



Triangular diagram of the system acetic acid / chloroform/ water



# Evaluation

## Evaluation (1/6)

### Result (1/3)

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

$$P + F = 5$$

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.



## Evaluation (2/6)

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### Result (2/3)

The following applies for a ternary system:

$$\chi_A + \chi_B + \chi_C = 1$$

where

$\chi_i$  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.

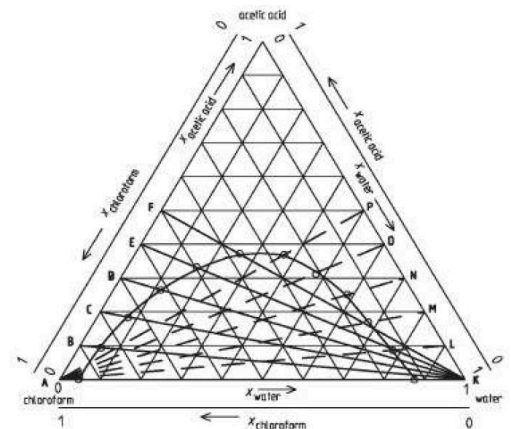
## Evaluation (3/6)

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### Result (3/3)

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).



Triangular diagram of the system acetic acid / chloroform/ water

## Evaluation (4/6)

What describes Gibb´s phase law?

Gibb's phase law describes the connection between a solid and a gas.

Gibb's phase law describes the relationship between the number of degrees of freedom  $F$  in a system, the phases  $P$  formed by the system and the one component, that is used in the system.

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.

✓ Überprüfen

## Evaluation (5/6)

What is the sum of phases and the degree of freedom in a three component system ( $K = 3$ ) ?

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

None of the answers is correct.

In a three component system ( $K = 3$ ), the sum of phases and the degree of freedom is 6.

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

✓ Überprüfen

## Evaluation (6/6)

Mark the correct statements.

- Both chloroform / acetic acid and water / acetic acid are completely miscible.
- None of the answers is correct.
- Chloroform / water are not miscible in any ratio.
- In a single phase system ( $P = 1$ ) under isothermic and isobaric conditions (= 2 degrees of freedom), two degrees of freedom remain.

✓ Überprüfen

Folie	Punktzahl/Summe
Folie 16: Gibb's phase law	0/1
Folie 17: Three component system	0/1
Folie 18: Miscible systems	0/3

Gesamtsumme  0/5

👁️ Lösungen

🔄 Wiederholen