Experimental Chemistry I

Basic Experiments
1-15
Protocol

2nd of March 1998
through
13th of March 1998

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Salzburg, April 4th 1998

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Appendix

Experiment 1: Day 1, 2nd of March 1998

Preparing Standard- and Reference-solutions (Acid/Base)

1.1 Purpose: Preparation of a *standardized acid* and a *standardized base* solution with an average concentration of c ≈ 1mol/L. The concentrations of these solutions are verified via titrations.

material: Burette stand with clamp
- 100mL graduated cylinder
- 250mL Erlenmeyer flask
- Pipette filler (Peleus) attached to a 25mL volumetric pipet AS-class
- 2 x 1L volumetric Florence flask
- 2 x 1L plastic bottles w/ tap
- 50mL burette w/ tripod and clamp
- 100mL beaker
- Glass funnel
- Magnetic stirrer
- Protection glasses
- Paper towels
- Marker pen (water proof)

chemicals: Deionized water
- Master Base / Acid :
  - ≈ 100mL (w=32%) NaOH
  - ≈ 100mL (w=32%) HCl
- Titrant base: (calibrator)
  - ≈ 100mL of 1.000M NaOH
- few drops of pH indicator: phenolphthalein (PP)

Procedure: Wash and rinse all utensils with deionized water. Clamp the burette onto the burette stand.

Preparation of Standardized Solution (c ≈ 1mol/L):

1. Calculate the volume of MASTER base solution required to prepare the Standardized Base Solution (see formula 1.1 & 1.2);
2. pipette the calculated volume into a 100mL graduated cylinder;
3. transfer this sample of MASTER base into a 1L volumetric flask and fill up the rest with deionized water;
4. seal and shake flask vigorously; store properly; mark flask with date, group, etc.; needed in experiment 8.
5. repeat steps 1 to 4 with MASTER acid to obtain the Standardized Acid Solution.

Note: Never pour any residual base/acid extracted from the MASTER base/acid back into the main storage bottle; use protection glasses at any time when handling acids or bases.

Results and Evaluation:

Volumes of MASTER base/acid needed to be extracted from the storage bottles:

Formula 1.1: (see appendix-formula for details)

$$c_{\text{HCl / NaOH}} = \frac{w_{\text{acid / base}} \cdot \delta_{W\,32\%}}{100 \cdot M_{\text{HCl / NaOH}}}$$

w, mass percentage [%]
ρ, density [g/L]
M, molar mass [g/mol]

Formula 1.2: ($c_1 \cdot V_1 = c_2 \cdot V_2$)

$$V_{\text{concentrated}} = \frac{c_{\text{diluted}} \cdot V_{\text{diluted}}}{c_{\text{concentrated}}}$$

c, concentration [mol/L]
V, volume [L]

Results: the following volumetric amount of concentrated acid / base is required (indicated in grey) to obtain a Standardized Solution with a molar concentration of c ≈ 1mol/L (1.0M)

standardized solution	w [%]	ρ* [g/L]	M [g/moL]	$c_{concentrated}$ [mol/L]	$c_{diluted}$ [mol/L]	$V_{diluted}$ [L]	$V_{concentrated}$ [mL]
HCl	32	1160	36.45	10.18	1	1	98.20
NaOH	32	1350	39.99	10.80	1	1	92.59

(*) density values obtained from data sheets provided

Ideally, for a strong acid- strong base titrations an indicator should have a sharp color change close to the stoichiometric point (S) of the titration, which is at pH = 7. However, the change in pH is so abrupt and the slope quite steep that even phenolphthalein can be used, which has a pH sensitive range of 8.2 to 10.

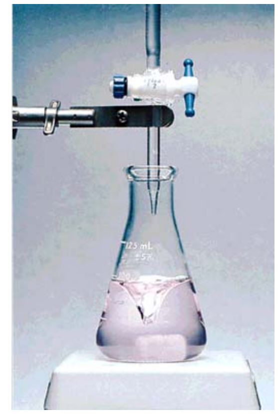
1.2 Titration (verification of results obtain from Experiment 1/1.1):

Purpose: Find the concentrations of the Standardized Acid / Base with the help of titration.

The reaction between a strong acid and a strong base can be basically considered as a **neutralization reaction**. In a neutralization reaction, an acid reacts with a base to produce salt and water:

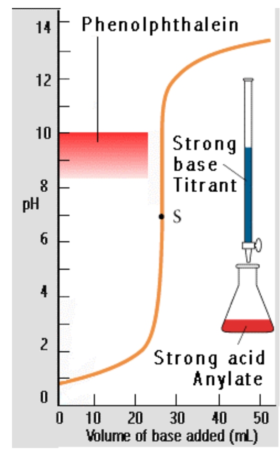
$NaOH(aq) + HCl(aq) \rightarrow 2H_2O(l) + Na^+(aq) + Cl^-(aq)$

An *indicator* enables detection of the **stoichiometric point** (S), the stage at which the volume of titrant added (with a given concentration) is exactly that required to neutralize the analyte (based on the stoichiometric relation between titrant and analyte).


Setup of Titration

Procedure: Clean 50mL burette with deionized water and

1. rinse burette with calibration solution (1.000M) NaOH and fill it up to zero-mark; close stopcock before filling with titrant and clamp burette onto the stand;
2. pipet 25ml of Standardized Acid into the 0.25L Erlenmeyer flask and add approx. 0.1L of deionized water;
3. add 1 drop of PP-indicator and magnetic rod into the flask; place Erlenmeyer flask and magnetic stirrer under the burette;
   Note: Add only one or two drops of indicator, so as not to upset the accuracy of the titration;
4. turn magnetic stirrer on and drip titrant into the analyte until change of color is permanent;
   Note: Observe the reaction closely to detect sudden changes in colour (to increase contrast, place white paper in-between flask and stirrer);
5. record the volume of calibration titrant consumed and calculate the concentration;
6. execute at least 3 titrations to determine a mean value; label flask with the concentration obtained;
7. seal volumetric flask containing the Standardized Solution and store properly; mark with the calculated concentration;
8. repeat steps 1 to 7 to determine the concentration of Standardized Base (where Standardized Acid with the calculated concentration is used as the titrant and Standardized Base in the Erlenmeyer flask is the analyte).



Titration curve highlighting the neutralization (**stoichiometric**) point S.

Results and Evaluation: Formula 1.2 has been used to determine the concentration of the individual acid / base:

Results of titration of Standardized Solution (Std) - (indicated in gray):

	Determination of Std.-Acid concentration				Determination of Std.-Base concentration			
	calibrating Titrant		Analyte		Titrant		Analyte	
trial	c_{NaOH} [mol/L]	V_{cNaOH} [mL]	V_{HCl} [mL]	c_{HCl} [mol/L]	c_{HCl} [mol/L]	V_{HCl} [L]	V_{NaOH} [L]	c_{NaOH} [mol/L]
1	1.000	24.60	25.00	0.984	0.988	25.00	25.20	0.980
2	1.000	24.70	25.00	0.988	0.988	25.00	25.20	0.980
3	1.000	24.80	25.00	0.992	0.988	25.00	25.20	0.980
averaged				0.988				0.980

1.3 Reference Dilution made from Standardized Acid and Base: Day 2, 3rd of March 1998

Purpose: Preparation of a dilution made of one part Standardized Acid / Base mixed with nine parts deionized water to obtain a reference acid / base solution of c ≈ 0.1mol/L (0.1M). These Reference Acid and Base Solutions are used in experiments 6 and 7.

Procedure:
1. pipet 100mL of Standardized Acid into a 1000mL volumetric flask;
2. add deionized water until the 1000mL mark is reached;
3. store the diluted reference acid in a tightly locked 1L plastic container;
4. label container with concentration (c ≈ 0.1mol/L or 0.1M), date, group, etc.;
5. repeated procedure (steps 1 to 4) is with the base;

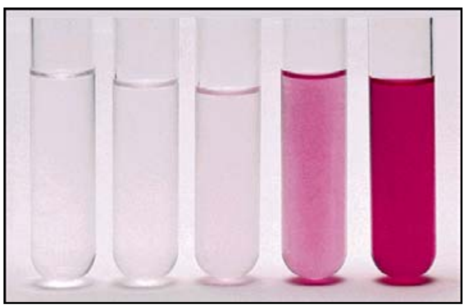
Results and Evaluation:

After having completed the dilution, both Reference Acid and Reference Base have approximately the concentration of 0.1mol/L; (using formula 1.2)

Results of dilution (indicated in gray):

	$V_{\text{Standard Sln}}$ [mL]	$c_{\text{Standard Sln}}$ [Mol/L]	$V_{\text{Reference Sln}}$ [mL]	$c_{\text{Reference Sln}}$ [mol/L]
HCl	100	0.988	900	0.0988
NaOH	100	0.980	900	0.0980

Index: Sln: Solution



PP-indicator is used as a visual aid to monitor changes in pH; it changes colour at pH of around ≥8.2 (pale pink) to 10 (dark red)

Experiment 2: Day 2, 3rd of March 1998
Precipitation Reaction - Recrystallization of PotassiumPerChlorate ($KClO_4$)

2.1 MnO_2 Elimination Reaction by filtration: a $KMNO_4$ sample provided by the tutors needs to be purified by removing the $KClO_4$ contaminant.

material: 250mL filter flask w/ filtervac neck ring (suction filtration)
Small Buchner filter
Water-jet evacuation pump w/ set of rubber hose
500mL Erlenmeyer flask
3 x 250mL beaker
500mL beaker filled w/ ice-water
Watch-glass (Ø 60mm)
Small glass funnel w/ glass wool
Small spatula
Magnetic stirrer w/ integrated heater
Desiccator with silica gel
Oven (max 200ºC)
Protection glasses
Marker pen
Paper towels
chemicals: Deionized water
Shredded ice
10g sample (¾ $KClO_4$ contaminated w/ ¼ $KMnO_4$)
≈100mL HCl (w=32%)

Purpose: A mixture of two different mole contents possesses different saturation characteristics. $KClO_4$ contaminated with $KMnO_4$ is dissolved in deionized water. In order to separate these two compounds, the mixture has to be deprived of impurities. These impurities are a reaction by-product, which forms when the mixture comes in contact with in water (Manganese-IV-Oxide, MnO_2) and can be eliminated when filtrating the precipitation product out of the suspension.

Unbalanced REDOX equation: (ON = oxidation num.)
$MnO_4^- + 2O^- \leftrightarrow MnO_2 + O_2$
RED: $MnO_4^- \rightarrow MnO_2$ (ON_{Mn} changes from +7 to +4)
OX: $2O^- \rightarrow O_2$ (ON_O changes from -2 to 0)
Half-Reactions: (separation of K^+ implies basic medium):
$3e^- + 4H^+ + MnO_4^- + 4OH^- \rightarrow MnO_2 + 2H_2O + 4HO^-$
simplified: $3e^- + 2H_2O + MnO_4^- \rightarrow MnO_2 + 4HO^-$
$2O^- \rightarrow O_2 + 4e^-$
Electron equalization:
$12e^- + 4MnO_4^- + 8H_2O \rightarrow 4MnO_2 + 16HO^-$
$6O^- \rightarrow 3O_2 + 12e^-$
Adding the two half-reactions:
$4MnO_4^- + 8H_2O + 6O^{2-} \rightarrow 4MnO_2 + 16HO^- + 3O_2$
extended with $12H^+$ and K^+ on both sides:
$4MnO_4^- + 8H_2O + 6O^{2-} + 12H^+ \rightarrow 4MnO_2 + 16HO^- + 12H^+ + 3O_2$
$4KMnO_4(s) + 8H_2O(l) \rightarrow 4MnO_2(s) + 4HO^-(aq) + 3O_2(g) + K^+(aq)$


Stripping of the MnO_2 from the $KClO_4$ / $KMnO_4$ mixture

Procedure: Rinse all utensils with deionized water;
- heat 250mL of deionized water in a 250mL beaker and keep warm;
- dissolve the 10g mixture ($KClO_4$ / $KMnO_4$) in a 150ml beaker, add a small quantity of the preheated water, add magnetic rod, and place on stirrer;
- while stirring bring solution to boiling point, keep adding preheated water until all of the precipitate has dissolved completely;

During the course of the dissolving process, MnO_2 as a by-product is formed, which must be removed;
- warm a funnel and filter by pouring preheated distilled water through the glass-wool popped funnel;
- start the actual filtration procedure with the heated $KMnO_2$ / $KMnO_2$-solution;
  Note: to avoid any loss of prime material, do not allow the filter-funnel to cool off; otherwise, crystalline $KClO_4$-precipitate instead of MnO_2 will be held back by the glass-wool; if a $KClO_4$ precipitate should form and be trapped by the glass-wool proceed by adding some hot water to the point that the prematurely formed precipitate re-dissolves again;
- reheat (boil) the filtered $KMnO_2$/$KMnO_2$-solution and proceed with procedure 2.2.

Results and Evaluation: Color of glass-wool in funnel changed from shiny white to a grayish brown. Drained filtered solution is left without any visible floating debris.

2.2 Separation of $KClO_4$ from $KMnO_4$ by repeated boiling:

Purpose: The filtrated solution obtained from procedure 2.1 is now ready to undergo another cycle of several loops of heating, cooling and filtration. While the solvent (deionized water) evaporates, heating the solution results in a gradual increase of solute concentration ($KClO_4$ + $KMnO_4$) in the solution.
The mixture with the super-saturated $KClO_4$ content can easily be removed from the dissolved $KMnO_4$ by filtration.

Buchner funnel used as a suction – filter device

Procedure: Precipitate $KClO_4$ and place in concave watch-glass;

- weigh watch-glass (dry & empty) and record its Tara-weight;

1. reheat mixture until crystalline-like film on surface of solution appears (indicating that solution is super-saturated and cannot hold more $KClO_4$ solute);
2. place beaker with salt-mixture in a beaker with ice-chilled water and allow to cool off, as a result of the rapid cooling process a coarse-sized $KClO_4$ precipitate will form;
   once cooled off, use a Buchner funnel device to "suction filter" the humid precipitate; doing so removes excess $KMnO_4$ solute; if any residual precipitate remains in the beaker, deplug sucking hose from the filter-flask, rinse beaker with the obtained effluent and repeat sucking procedure;
   Note: Never turn off the faucet while filtering is still taking place; otherwise, the lower-than-air pressure conditions within the flask will suck up water from the faucet through the pipe into the flask;

- repeat procedure with $KMnO_4$ runoff at least three times; this guarantees that residual $KMnO_4$ still present in the runoff can be eliminated; to do so start with:
  i) take the crystalline filtrate, dissolve again with some of the warmed deionized water, and repeat again steps 1 & 2;
  i) any debris of Manganese Oxide (MnO_2) left in the Buchner-filter can be eliminated with concentrated HCl;
  i) with every step in purification, the crystalline precipitate shifts from pink to whitish;
- after the final purification-step, place the crystalline $KClO_4$ onto the watch-glass, dry it in the oven or store it in the desiccator for final evaluation (weighing) by the tutors.

Results and Evaluation: the obtained mass of the extracted and precipitated $KClO_4$ amounts to (indicated in grey):

	watch-glass	glass + filtrate (wet)	glass + filtrate (dry)	$KClO_4$ filtrate (dry)
mass [g]	36.96	43.92	43.83	6.86

The weight of dry filtrate obtained after three filtration cycles represents more or less a compromise between purification and loss in mass of the final precipitate. The more filtrations are executed the more prime material is lost during this process. Based on the original amounts of $KClO_4$ used (information obtained from tutors), we achieved an overall recuperation level of about 69%.

Experiment 3 - Enrichment of Ethanol (C_2H_5OH): Day 2, 3rd of March 1998

3.1 Dehydration of Ethanol with CalciumOxide (CaO):

Purpose: In the 1st step of this experiment, high grade EtOH is enriched to a level of purification of w = 95%. A further decrease in the water-content to <u>at least 99%</u> can only be achieved chemically by introducing CaO granulate which absorbs residual H_2O:

$CaO(s) + H_2O(l) \leftrightarrow Ca(OH)_2(s)$

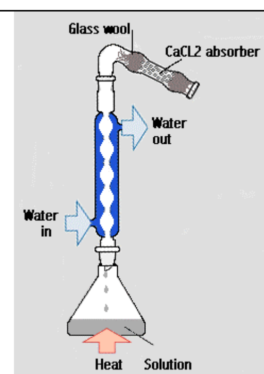
Because fractional distillation (a procedure used for separating components with different boiling points) provides high purification but low yield, the Reflux-technique is used instead. This closed-loop distillation procedure prevents evaporation of ethanol, while at the same time allowing effective circulation of distillate and water absorption (obtained via chemical means). A hygroscopic water-vapor absorber ($CaCl_2$) attached on top of the condenser prevents that ambient humidity (air) interferes with this enrichment process (EtOH with a mass percentage >96% is highly azeotropic).

material: Stand w/ clamps
- 250mL filter flask w/ filtervac neck ring (suction filtration)
- Large Buchner funnel
- Water-jet evacuation pump w/ set of rubber hoses
- 500mL Erlenmeyer flask w/ stopper
- 1L Erlenmeyer flask w/ stopper
- Water cooled distilling apparatus
- Water cooled dockable Reflux (Dimroth) condenser
- Drier dockable onto condenser
- Large mortar and pestle
- Small spatula
- Stopper thermometer (max. 150°C)
- Magnetic stirrer w/ hot-plate
- 2 Lab-jacks
- Digital single-pan balance
- Bunsen burner
- Oven (max 200ºC)
- Stopcock silicon grease
- Protection glasses
- Paper towels
- Laboratory film (Parafilm - M)

chemicals: Deionized water
- Acetone for cleaning purposes
- 20g Calcium Chloride granula $CaCl_2$ (for drying, w = 93%)
- ≈70g Calcium Oxide from marble CaO (small lumps)
- ≈3g of metal. Mg
- ≈0.5g of I_2 (if liquid: few drops)
- 350mL Ethanol C_2H_5OH (w ≈95%)
- ≈1g of water-free Copper Sulfate $CuSO_4$ (w = 95%)

Procedure: Prior to usage, <u>wash all glassware thoroughly, rinse with acetone and dry in oven</u>. Assemble reflux-apparatus on stand w/ clamps (incl. $CaCl_2$ drier), magnetic stirrer and lab-jack (see sketch below).
Note: seal absorber ($CaCl_2$) stage with parafilm if glassware is not used immediately (exposure to air reduces absorptive capacity of vapour); for air-tightness, do not forget to seal joints with grease; screws holding glassware must not be tightened!

- grind CaO granulate w/ mortar & pestle (<5mm);
  Note: wear protection glasses (must be done under aspirator as CaO is very corrosive when moist).
- place ≈70g CaO powder into the 500mL flask, add magnetic rod, attach reflux apparatus onto the flask and place system onto hot plate / stirrer;
- through the top of apparatus (w/ unattached $CaCl_2$ stage) pour the 350mL EtOH into flask;
- attach water hoses to the cooling system (open faucet), heat up and boil for <u>2 hours</u>; make sure that evaporating and condensing ethanol drips back into the flask (observe condenser, if necessary readjust thermostat);
- afterwards, allow to cool off by lowering hot plate on lab jack – leave stirrer on (at this point, EtOH has reached 95%, it shows azeotropic properties); finally remove and seal flask);
- suction-filter solution w/ Buchner funnel to remove CaO;
- store the purified EtOH in an <u>airtight</u> 500mL Erlenmeyer flask;
  Note: If the faucet is turned off while filtering; the negative pressure w/n flask will suck water from the pipe into the flask.

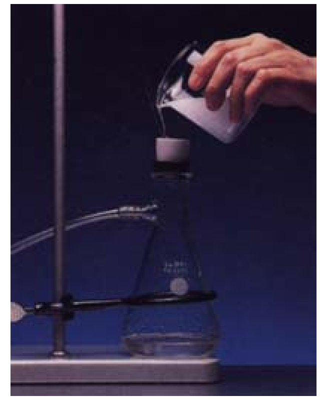


Reflux-technique used in the enrichment procedure

Results and Evaluation: Unfortunately, while sucking the purified EtOH through the Buchner funnel, the jet-attachment of the water pipe broke, causing tap-water to be sucked into the flask, diluting the enriched EtOH....☹

3.2 Dehydration of EtOH with Magnesium & Iodine (100% water-free): Day 3, 4th of March 1998

Purpose: To further boost the purity-level of EtOH to almost 100%, the dehydration procedure has to be performed with different means. Because distillation techniques will not work beyond alcohol contents >96% (mass percentage), a stoichiometric method is used instead. The remaining water in solution is forced to react with metallic Mg. To lower the activation energy, iodine is added to trigger the reaction:



Buchner funnel used as a suction –filter device

$C_2O_5OH(l) + Mg(s) + I_2(s) \rightarrow Mg(OC_2O_5)_2(aq) + 2HI(l)$

I. $Mg(OC_2O_5)_2(aq) + H_2O \leftrightarrow Mg(OH)_2(aq) + H_2(g)$

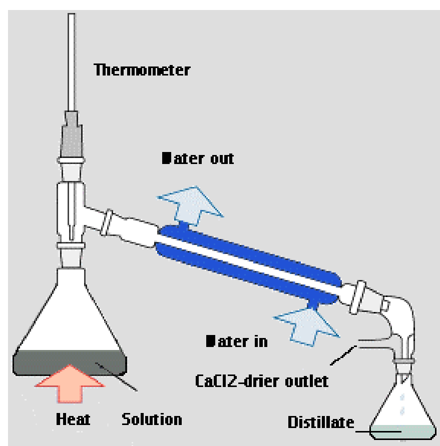
$Mg(OH)_2(aq) + 2HI(l) \rightarrow MgI_2(s) + 2H_2O(l)$

II. $Mg(OC_2O_5)_2(aq) + 2H_2O(l) \rightarrow Mg(OH)_2(aq) + \mathbf{2C_2O_5OH}(l)$

This is the n followed by a distillation procedure.
The volatilised EtOH distillate passing through the condenser is collected in the separately attached flask.
The Mg-hydroxide will remain as a precipitate in the heated Erlenmeyer flask.

Procedure: Reassemble reflux-apparatus on stand w/ clamps as shown in the sketch of the previous page (incl. drier filled with $CaCl_2$), magnetic stirrer and lab-jack;

- place ≈3g of solid Mg and I_2 into the flask and add 50mL of the 99% EtOH (solution turns brownish);
- start the reaction by heating gently under constant motion (magnetic stirrer) and pour the remaining refined EtOH through the Reflux apparatus once the brownish colour has faded;
- turn on the cooling system, heat to boiling point and keep simmering for at least 2 hours;
- before disassembling the Reflux apparatus, remove concentrated EtOH with the grayish precipitate and seal properly (with laboratory film);
- set up distillation apparatus (incl. stopper-thermometer and the $CaCl_2$-tube attached to the 300ml Erlenmeyer receiving flask – see sketch below);
  Note: Seal joints with stopcock grease; make sure that $CaCl_2$ drier is not popped w/ stopper or film;
- keep boiling under constant motion (magnetic stirrer) until at least 200mL EtOH has been collected in the receiving Erlenmeyer flask.
  Note: Do not leave the distillation procedure unattended; pure EtOH is highly inflammable;



Enrichment procedure to obtain pure EtOH

To make sure that no water is left in the distillate, a final test with $CuSO_4$ is done:

- desiccate a pinch of $CuSO_4$ (with a Bunsen-burner under aspirator) until bluish color is gone;
  Note: make sure that no volatile liquids are located within the aspirator (beakers with unknown contents may explode);
- drip a few drops of the distillate onto the pale $CuSO_4$; if no change in color occurred, the distillate can be considered water-free (counter-test with a few drops of water to enforce a colour change);
- keep stopper onto flask and seal with laboratory film to avoid evaporation or absorption of humidity from the ambient (100% EtOH is extremely hygroscopic); this EtOH will be needed for experiment 10 and 13.

Results and Evaluation: Due to the diluted sample caused by the defective water-jet pump, we have been given a sample of 99% purified EtOH and were thus able to obtain 250mL of concentrated and dehydrated EtOH (100%)....☺

Experiment 4: Day 3, 4th of March 1998

Complexometry - Extraction of $FeCl_3$ from a watery solution:

4.1 Extraction of $FeCl_3$ from an acidic solution into an organic phase:

Purpose: An unknown iron-containing compound ($FeCl_3$) should be extracted and eventually quantitatively determined. To do so the salt is dissolved in 8M strong HCl (high in demand of Cl^-):

$$FeCl_3(s) + 3HCl(aq) \leftrightarrow H_3FeCl_6$$
$$H_3FeCl_6 \leftrightarrow 3H^+ + [FeCl_6]^{3-}$$

This complex Chloroferrate-III compound dissolves readily in a hydrophilic solution. By shaking the mixture vigorously, (decreasing size of micelles, result in a net increase of surface area) $[FeCl_6]^{3-}$ attaches easily onto the polar end (O of the Keto-group) of the MIBK (methyl-isobutyl-keton), literally "encapsulating" the compound.
The fact that MIBK possesses a methyl and an isobutyl fatty chain attached to the Keto-group as well, helps to swap the $[FeCl_6]^{3-}$ from the watery to the hydrophobic phase.
In a reverse step, the complex compound is lateron transferred from the fatty back into the hydrophilic watery phase (4.2).

material: Ring stand w/ clamps
- 10mL buret
- 300mL wide-mouthed Erlenmeyer
- 500mL Separator flask w/ stopper
- 3 x 100mL volumetric Florence flask w/ glass stopper
- 150mL beaker
- 50mL beaker
- 30mL beaker
- Pipette filler (Peleus) along with 25mL volumetric pipette
- Flat-pan balance
- Protection glasses
- Paper towels
- Pasteur pipette

chemicals: Deionized water
- ≈15g Iron(III) Chloride pure in small lumps $FeCl_3$
- 20ml of 10M Hydrochloric Acid HCl (w = 32%)
- ≈60mL MIBK $C_6H_{12}O$
- a pinch of Ammonium Thyiocynate GR, NH_4SCN (w = 99%)
- pH indicator paper
- 5g Sulfosalicylic Acid
- 100ml Titriplex-III for metal titra-tion Na_2-EDTA-$2H_2O$ (0.1M)
- a few drops of HNO_3 (could be necessary for experiment 4.3)

Procedure:
- Dissolve $FeCl_3$ in 20mL HCl (w = 32%) in a 50mL beaker, to produce a dark yellowish solution;
- add the calculated volume of water (≈5.5mL) to obtain an 8M HCl; and wait until completely dissolved (using formula 4.1 and 4.2);
- transfer solution into separator flask and add 15mL of MIBK;
- shake separator flask vigorously;
  Note: Vapour pressure inside the flask increases drastically; therefore, ventilate flask from time to time;
- wait until the phases are clearly separated;
- drain off the watery phase into a 30mL beaker, whereas the yellowish hydrophobic (MIBK) phase is collected in a separate 100ml Erlenmeyer flasks;
- pour the watery phase back into the separator flask along with another 15mL MIBK, shake well, ventilate, and eventually allow phases to separate again;
- drain off the watery phase into a 30mL beaker, while the MIBK phase is added to the first sample of the 100ml Erlenmeyer flasks;
- repeat the separation procedure for a third time.

Separator flask

4.2 Testing for any residual iron within the watery phase:

Procedure: This test should make sure that no iron residuals are left in the aqueous phase (all iron traces should have been translocated into the hydrophobic phase);

- dissolve a pinch of NH_4SCN into the aqueous phase obtained from the phase-separator; (there should be no visible reaction whatsoever);
- to make sure that the probe was working, add some $FeCl_3$ which stains the solution dark red;

4. 3 Transferring the Iron compound back to the watery phase:

Purpose: Extracting the Chloroferrate-III complex back to a hydrophilic medium causes the compound to split into its ionic constituents (Fe^{3+} and Cl^-) which are easily soluble in water. This watery solution is later on needed to determine the amount of iron and chloride in a titration procedure.

Procedure: in order to achieve hydrolysis of the ionic constituents,

- the yellowish and hydrophobic MIBK phase is placed back into the separator flask and mixed with 20mL deionized water; shake vigorously and ventilate from time to time;
- allow the two phases to separate (watery phase turns yellowish; MIBK-phase fades out);
- collect the watery phase in a 100mL volumetric flask, MIBK phase remains in the separator flask;
- add further 20mL deionized water to the separator flask; repeat procedure three to four times;
- fill up the extracted watery phase with deionized water until the 100ml mark is reached; seal and store properly (to be used for experiment 12).

Results and Evaluation of phase separation: In order to determining the volume required to dilute concentrated HCl (w = 32%) to obtain a desired concentration of 8M, the following calculations have to be made:

Formula 4.1: (for details see appendix-formula)

$$c_{HCl} = \frac{w_{acid / base} \cdot \delta_{W\,32\%}}{100 \cdot M_{HCl}}$$

w, mass percentage [%]
ρ, density [g/L]
M, molar mass [g/mol]

Formula 4.2: used to obtain a 8M HCl ($c_1 \cdot V_1 = c_2 \cdot V_2$)

$$V_{diluted} = \frac{c_{concentr.} \cdot V_{concentr.}}{c_{diluted}}$$

c, concentration [mol/L]
V, volume [L]

The volumetric differences of the initial and the final concentration yields the volume of deionized water needed. The dilution lowers the initial concentration of the HCl to the demanded value.

Results: the required amount of water for this diluted acid concentration is (indicated in grey):

reference solution	concentrated $HCL_{32\%}$					diluted HCL		
	w [%]	ρ* [g/L]	M_{HCl} [g/mol]	$c_{conc.}$ [mol/L]	$V_{conc.}$ [mL]	$c_{diluted}$ [mol/L]	$V_{diluted}$ [mL]	V_{water} (difference) [mL]
HCl	32	1160	36.46	10.18	20.00	8.00	25.49	5.490

*) density values obtained from data sheets, see appendix

Staining test (testing for any residual iron within the watery phase): the NH_4SCN probe did not stain the acidic solution, indicating that a high degree of separation has been reached; cross-checking with a $FeCl_3$-sample yielded the expected staining-results.

Extraction (transferring the Iron-compound back to a watery phase): Performing the last step (refilling with deionized water) acetone has been mistakenly used to dilute the watery solution. Consequently, the mistakenly-diluted sample has been left open to allow evaporation of acetone. Only after two days it was filled up with deionized water to the 100mL mark. Nonetheless, the entire separation procedure was repeated to obtain a second sample.

4.4 Titration of dissolved Iron with EDTA (Edetinacid):

Purpose: Fe^{3+} -ions are bound to the EDTA when brought together. A high acidic medium is needed to bind the iron ions to the EDTA. The three positive charges ($^{3+}$) make this complex very stable. The high Stability Constant reflects this stability; therefore, only a low amount of EDTA as titrant is needed to bind all Fe-ions in solution. An indicator is added to show the presence of dissociated Fe^{3+} -ions (dark reddish-blue). Once all Fe^{3+} ions are fixed by EDTA, the less stable indicator-complexes are broken up, gradually leading to a change of color (solution becomes pale, yellowish). Although the detection of Fe with EDTA is not specific to Fe alone, it is sufficient for this experiment (elements like Zn, Mn, Co, Ni, Cu are not included in the sample given).

Procedure: Rinse all utensils with deionized water and clamp 10mL burette onto the stand:

- rinse burette with titrant (EDTA, c = 0.1mol/L) and fill it up to zero-mark; in case bubbles are formed during the filling process, use a Pasteur pipette to squeeze trapped air out of the column.

Preparation of analyte:

- pipet 10mL of Isobuthyl-Methyl-Keton in the 300mL wide-mouthed Erlenmeyer flask and add 100mL of deionized water;
- verify the pH of the extracted sample with indicator paper; if too high (>3), add a drop ofHNO_3 and check again until around or below 2.5;

Preparation of indicator:

- weigh 5g of Sulfosalicylic Acid into a 150mL beaker and fill up with 100mL deionized water;
- add 1mL to the 300mL Erlenmeyer containing the Isobuthyl-Methyl-Keton sample;

Titration:

- start titration until colour changes from dark reddish-blue to the yellowish background hue;
- record the consumed volume of titrant (repeat titration at least three times);
- average the titration results and calculate the iron content (use formula 4.3); every mL EDTA consumed, fixes the equivalent of 5.585mg of ferric-ions.

Results and Evaluation of phase separation:

Both samples (normal and accidentally treated acetone solution) are examined for their iron content. Ironically, the acetone- treated solution yielded far better results than the correctly prepared sample:

Formula 4.3: (for details see appendix-formula)

$$m_{Fe} = \frac{V_{EDTA} \cdot 55.85 \cdot E^{3} \cdot M_{FeCl_3 \cdot 6H_2O}}{V_{diluted} \cdot M_{Fe}}$$

V, volume	[L]
M, molar mass	[g/mol]
m, mass	[g]

Results: the calculate amount of iron in the sample is (indicated in grey):

	normal sample	acetone treated		burett e			repeated sample	acetone treated
	V_{EDTA} [mL]	V_{EDTA} [mL]	$V_{diluted}$ [mL]	V_{total} [mL]	M_{Fe} [g/mol]	$M_{FeCl3 \cdot 6H2O}$ [g/mol]	m_{Fe} [mg]	m_{Fe} [mg]
1st titration	4.43	4.95						
2nd titratn.	4.43	4.94						
3rd titratn.	4.46	4.92						
averaged	4.43	4.937	1	10	55.85	270.3	1.335	1.307

Experiment 5: Day 4/5: 5th/6th of March 1998

Redox Reaction - Oxidation of Zinc to Zincoxide (ZnO):

material: Porcelain dish (∅ 60mm)
- Crucible tongs
- Clay covered wire-triangle
- 15mL beaker
- Bunsen burner
- Vacuum Desiccator (w/ Silica-gel and $CoCl_2$ as humidity indicator)
- Digital single-pan balance
- Protection glasses
- Latex gloves
- Paper towels
- Pasteur pipette

chemicals:
- ≈1-2g of pure metallic Zn
- ≈10ml Nitric Acid HnO_3 (w = 65%)

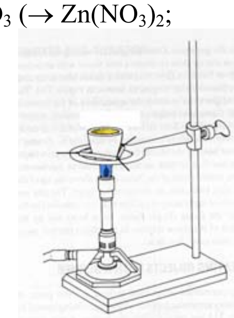
5.1 Dissolving metallic Zn in HNO_3: Mass determination of Zinc in ZincOxide.

Purpose: An unknown quantity of metallic Zinc, provided by the tutors, is dissolved in nitric acid, scalded, and the resulting zinc-oxide weighted:

$$Zn(s) + 2HNO_3(aq) \rightarrow Zn(NO_3)_2(aq) + H_2(g)$$
$$Zn(NO_3)_2(aq) \rightarrow(\text{approx.}1000°C)\rightarrow ZnO + N_2O_5(g)$$
$$N_2O_5(g) \rightarrow 2NO_2(g) + \tfrac{1}{2}O_2(g)$$

The amount of Zn is stoichiometrically calculated from ZnO and compared with the amount of Zn given by the tutors.

Procedure: Wash porcelain dish thoroughly with deionized water; place empty porcelain dish onto Bunsen burner;
- to eliminate residual weight due to absorbed humidity, desiccate porcelain dish for approx. 20mins under aspirator;
- use crucibles to place empty porcelain place in Desiccator and allow to cool off in a water free environment for another 20mins;
  Note: At the beginning, open ventilation valve of Desiccator to allow pressure compensation;
- place the solid Zn-sample into the porcelain dish and add approx. 5mL HNO_3 ($\rightarrow Zn(NO_3)_2$;
  Note: degassing of the extremely toxic brownish N_2O_5 must be done under aspirator, ***do not inhale it***; make sure that no volatile liquids are placed within aspirator; wear protection glasses and gloves (HNO_3 is a very corrosive acid!);
- if reactions slows down leaving undissolved Zn, add extra HNO_3;
- carefully heat up porcelain to desiccate solution; do not boil solution as bubbling of solution results in a loss of substance....☹;
- once the liquid phase has boiled off, dehydrate for another 20mins under Bunsen burner;
- use crucibles to place dish in Exsiccator to cool down (extra 20mins);
- weigh the porcelain with ZnO condensate, subtract the mass of the porcelain dish to and calculate the Zn-content in the residual ZnO.



Desiccation procedure using a porcelain dish and a Bunsen burner

Results and Evaluation of Extraction:

Due to excess temperatures used during the heat-treatment, more than 3% ZnO was lost. As a result, and due to extensive mismatch between the original mass and the obtained mass, the experiment has been repeated for several times....☹.....before achieving acceptable results.

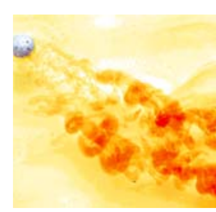
Formula 5.1: (for details see appendix-formula)

$$m_{Zn} = \frac{(m_{porcelain+ZnO} - m_{empty\ porcelain}) \cdot M_{Zn}}{M_{ZnO}}$$

M, molar mass [g/mol]
m, mass [g]

Results: the calculate amount of iron in the sample is (indicated in grey):

$mass_{empty\ porcelain}$ desiccated [g]	$mass_{porcelain+ZnO}$ desiccated [g]	m_{ZnO} [g]	M_{ZnO} [g/mol]	M_{Zn} [g/mol]	$mass_{Zn}$ [g]
36.91	38.49	1.580	81.40	65.40	1.269



Zn reacting with HNO_3

Experiment 6: Acid-Base Reaction: Day 4: 5th of March 1998
Determining the concentration of diluted Acetic Acid (HAc):

material: Stand w/ Burette-clamp
- 50mL burette (depending upon the demand of titrant)
- 100mL volumetric-Florence flask
- 200mL wide-mouthed Erlenmeyer flask
- Pipette filler (Peleus) with 0.1L volumetric pipette AS-class & 2mL volumet. pipette AS-class
- 100mL beaker
- Magnetic stirrer
- Protection glasses
- Paper towels
- Marker pen

chemicals: Deionized water
- Acetic acid CH_3CO_2H (HAc, w ≈ 7%) analyte from tutors
- ≈50mL of Reference Base: NaOH from exp. 1; c = 0.0980mol/L
- few drops of phenolphthalein indicator (PP)

6.1 Estimating the demand of Titrant needed:

Purpose: The aim of this experiment, it is to calculate the approximate amount of titrant needed and based on that estimation to choose an adequate burette size to execute the titration described under 6.2.

Procedure: It is assumed that the Acetic acid sample (HAc) given by the tutors does indeed have a mass percentage of 7% (which roughly corresponds to a molar concentration of 1mol/L). The approximated volume of titrant required is calculated on the basis of formula 6.1. Based on its result, a suitable burette size can be chosen for this titration.

Results and Evaluation:
As indicated by the mathematical procedure below, approximately 24mL of titrant will be needed. Therefore, a burette size of 50mL is best suited to execute the titration shown under 6.2

Formula 6.1: (see appendix-formula for details)

$$c_{HAc} = \frac{w_{HAc} \cdot \rho_{HAc}}{100 \cdot M_{HAc}}$$

w, mass percentage [%]
ρ, density [g/L]
M, molar mass [g/mol]

Formula 6.2: ($c_1 \cdot V_1 = c_2 \cdot V_2$)

$$V_{NaOH} = \frac{c_{HAc} \cdot V_{HAc}}{c_{NaOH}}$$

c, concentration [mol/L]
V, volume [L]

Results of estimation (indicated in gray):

estimated			Analyte		Titrant	
w_{HAc} [%]	ρ_{HAc}* [g/L]	M_{HAc} [g/mol]	c_{HAc} [mol/L]	V_{HAc} [mL]	c_{NaOH} [mol/L]	V_{NaOH} [mL]
7	1000	60.05	1.166	2	0.098	23.79

(*) density values obtained from data sheets, see appendix

6.2 Executing the Titration:

Purpose: Calculation of the accurate value of the mass percentage (w) contained in the analyte provided by the tutors:

$CH_3CO_2H + NaOH \rightarrow NaCH_3CO_2 + H_2O$

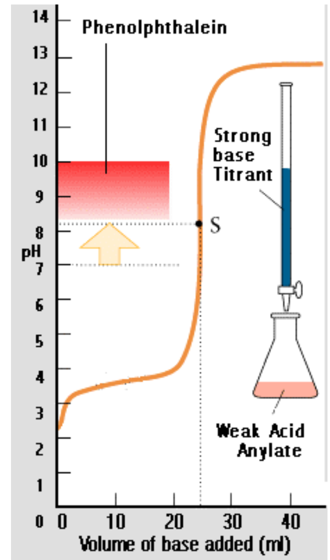
shorthand notation:

$HAc + NaOH \rightarrow NaAc + H_2O$

i.e.: 1mole of NaOH consumes 1mole of HAc

Procedure: Rinse all utensils with deionized water and clamp burette onto the stand;

- rinse 50mL burette with 0.1M reference base and fill it up to zero-mark;
- fill up the 100mL volumetric flask containing the 2mL HAc sample (w ≅7%, provided by the tutors) with deionized water; shake well;
- pipet 50mL of the sample into a 200ml Erlenmeyer flask along with magnetic rod and place onto stirrer; add 50mL of deionized water;
- add two drops of PP-indicator;
- turn stirrer on and start titration until the indicator changes from colorless to pink;
- record the consumed volume of titrant; for accuracy, repeat titration three times and average amount of titrant;
- calculate the concentration and exact mass percentage of the HAc (analyte) using formula 6.3 and 6.4;



Titration with the 0.1M Reference Base

Results and Evaluation: Misinterpreting the density of the weak acid, mistakenly, prompted us to repeat preparation of titrant (0.1mol/l base) as well as titration for a second time, until the error in the density-calculation has been realized.

Formula 6.3: ($c_1 \cdot V_1 = c_2 \cdot V_2$)

$$c_{\text{dil.HAc Analyte}} = \frac{c_{\text{NaOH Titrant}} \cdot V_{\text{NaOH Titrant}}}{V_{\text{diluted HAc Analyte}}}$$

c, concentration [mol/L]
V, volume [L]

Formula 6.4: (see appendix-formula):

$$w_{\text{dil. HAc Analyte}} = \frac{100 \cdot c_{\text{dil.HAc Analyte}} \cdot M_{\text{dil.HAc}}}{\rho_{\text{diluted HAc Analyte}}}$$

w, mass percent. [%]
ρ, density [g/L]
M, molar mass [g/mol]

Results of the mass percentage estimation (indicated in grey):

Titration	NaOH Titrant V_{NaOH} [mL]	c_{NaOH} [mol/L]	Diluted HAc Analyte V_{HAc} [mL]	c_{HAc} [mol/L]	M_{HAc} [g/mol]	ρ_{HAc}* [g/L]	w_{HAc} [%]
1st	24.40						
2nd	24.57						
3rd	24.53						
averaged	∑ = 24.50	0.098	2.000	1.201	60.05	1000	7.209

(*) density values obtained from data sheets, see appendix

The 7.2% density value was found to match perfectly with the original substance obtained from the tutors.

Experiment 7: Buffer System - Acid-Base Reaction: Day 5: 6th of March 1998

7.1 Calibration of electronic pH Meter and determination of pH:

Purpose: In order to perform a flawless titration and determination of pH (experiment 7.2), it is necessary to get acquainted with an electronic pH-meter. For this purpose the Reference Acid / Base from experiment 1 are used.

Procedure: Becoming familiar with the electronic pH-meter;

- rinse pH-sensor with deionized water, dry gently, turn on the meter, and dip electrode into the technical buffer (pH = 4.01) - wait until calibration of 1st reference point is completed (≈1min);
- rinse electrode again, dry gently, and dip into the 2nd technical buffer (pH = 7.00) until 2nd calibration mode is terminated (≈1min);
- rinse and dry electrode again and calibrate with 3rd technical buffer (pH = 10.00); rinse, dry, and place sensor in the KCl-filled storage-tube provided;
  Note: Store electrode always vertically; unplug ventilation stopper (if any) on electrode during use.
- extract 5mL from the Reference Acid and dilute with deionized water in a 250mL volumetric flask; determine the pH electronically - wait until read out is stable; rinse again after use, gently dry electrode and store in KCl-test-tube (test-tube rack);
- repeat previous procedure with Reference Base as well; extract 5mL, dilute, and measure its pH; rinse, gently dry and store the sensor again in KCL-solution.

material: Stand w/ Burette-clamp
50mL burette (depending upon the demand of titrant)
100mL volumetric-Florence flask
3 x 0.25L volumetric-Florence flask
Pipette filler (Peleus)
5mL volumet. pipette AS-class
50mL volumet. pipette AS-class
300mL beaker
100mL beaker
Test-tube & Test-tube rack
Automatic burette dispenser
Electron. pH-Meter w/ sensor stored in test-tube (KCL-solution)
Magnetic stirrer
Digital single-pan balance
Protection glasses
Paper towels
Marker pen

chemicals: Deionized water
Technical buffer pH 4.01
Technical buffer pH 7.00
Technical buffer pH 10.00
≈5mL of Acetic Acid glacial CH_3O_2H (HAc, w = 100%)
≈0.5g crystalline SodiumAcetate CH_3CO_2Na (NaAc, w = 99%)
≈110mL of Reference Acid: HCL from exp. 1; c = 0.0980mol/L
≈110mL of Reference Base: NaOH from exp. 1; c = 0.0988mol/L

Results of electronic pH measurement:

	reference acid c = 0.0980mol/L	reference base c = 0.0988mol/L
pH	2.43	11.46

Although a cross-check with this buffer should be executed with the proper reading, the technical buffer of pH10 did not yield any useful results at all. The exact reason for the mismatch could not be found, since tutors discouraged us using this particular buffer....

pH-measurement with electronic meter

7.2 Determining the buffer-capacity of a HAc / NaAc solution:

Purpose: Two buffer systems are tested for their buffering characteristics (titration is performed with Reference Acid / Base from experiment 1):
Buffer 1 consists of equal concentration (1:1) of acetic acid (HAc) and sodium acetate (NaAc).
Buffer 2 consists of 1/10th of the HAc, but the same NaAc concentration as buffer 1 (1:10).
The effects of titration are followed with a pH-meter and recorded to obtain a chart showing the characteristics of this particular buffer system. The reference acid/base are used for both buffers.

Procedure: As concentrated SodiumAcetate is solid, preparation of the NaAc solution is easiest by weigh the required amounts to obtain a 20mM solution;

- calculate the exact weight of NaAc, and the volume of concentrated HAc needed for both buffer systems (formula 7.3 with $V_{NaAc\text{-}dil} = 0.25L$ and c = 0.02mol/L);
- dilute calculated amount of NaAc (approx. 0.41g) w/ deionized water in 250mL volumetric flask;
- pipet the calculated volume of HAc (100%, approx. 1.14mL) into a 100mL volumetric flask (use automatic Burette) and dilute with deionized water[1];
- pipet 10mL of the diluted HAc into a 250mL volumetric flask; further dilute it with deionized water (till to the mark);

Set-up of titration utensils:

1. rinse and fill the 50mL burette with Reference Base for the 1st titration (≈0.1M NaOH as titrant);
2. mount pH electrode with a burette clamp onto stand;
3. use lab-jack and magnetic stirrer;

Preparation of **buffer #1**: (HAc and NaAc both with c = 0.02mol/L)

4. as for the analyte, pipet 50mL HAc and 50mL NaAc into a 300mL beaker;
5. execute 1st titration (best in 1mL steps - wait after each dose, until reading of pH-meter is stable);
6. record pH, volume of titrant consumed and temperature of analyte (draw a chart);
7. make a new buffer 1 and repeat titration (steps 1 to 6) with ≈0.1M HCl as titrant;

Preparation of **buffer #2**: (HAc with c = 0.002mol/L, and NaAc with c = 0.02mol/L)

1. for the 3rd titration rinse and fill the 50mL burette with the Reference Base (≈0.1M NaOH);
2. pipet 25ml of HAc (c = 0.02mol/L) into a 250mL volumetric flask and dilute w/ deionized water;
3. pipet 50mL of the diluted HAc and 50mL of NaAc into a 300mL beaker;
4. execute 3rd titration (best in 1mL steps - wait until read out of pH meter is stable);
5. make a new buffer 2 and execute 4th titration (repeat steps 1 to 4) using ≈0.1M HCl as titrant;

Results and Evaluation: Having diluted the titrant mistakenly to 1/10th of the given value forced us to repeat the first two titrations using the buffer #1. (see appendix - data sheet, for the detailed list stating each single datum during titration).
([1]) left without a 1L volumetric flasks, the obtained amount has been diluted to 1/4th to obtain the same concentration using only a 250mL volumetric flask; i.e. final volume of HAc is: **286.0** µL.

Formula 7.1: (see appendix-formula for details)

$c_{conc.HAc} = \dfrac{w_{HAc} \cdot \rho_{HAc}}{100 \cdot M_{HAc}}$	w, mass percentage ρ, density M, molar mass	[%] [g/L] [g/mol]

Formula 7.2: ($c_1 \cdot V_1 = c_2 \cdot V_2$)

$V_{conc} = \dfrac{c_{diluted} \cdot V_{diluted}}{c_{concentrated}}$	c, concentration V, volume	[mol/L] [L]

Formula 7.3: (for details see appendix-formula)

$m_{NaAc} = c_{NaAc} \cdot V_{NaAc} \cdot M_{NaAc}$	c, concentration V, volume	[mol/L] [L]

Results of calculation of NaAc (indicated in gray):

$c_{NaAc\text{-}dil.}$ [mol/L]	$V_{NaAc\text{-}dil}$ [mL]	M_{NaAc} [g/mol]	m_{NaAc} [mg]
0.02	0.250	82.03	410.2

Results of calculation of HAc (indicated in grey):

Titrant						Anylate		
c_{NaOH} [mol/L]	c_{HCl} [mol/L]	w_{HAc} [%]	ρ_{HAc}* [g/L]	M_{HAc} [g/mol]	$c_{HAc\text{-}con}$ [mol/L]	$c_{HAc\text{-}dil.}$ [mol/L]	$V_{HAc\text{-}dil}$ [mL]	$V_{HAc\text{-}con.}$ [mL]
0.0980	0.0998	100	1050	60.05	17.49	0.02	0.250	1.140

(*) density values obtained from data sheets, see appendix

7.3 Calculating the pH of buffer-1 and buffer-2:

Self-dissociation of **Buffer #1**: When mixing 50mL HAc (0.02M) with 50mL NaAc (0.02M) the overall volume of the solution changes, lowering the concentrations of the individual components to (0.01mol/L); see formula 7.2;
these new concentrations (using formula 7.4, 7.5, and 7.6) yield the pH of the acetate-solution;

Buffer #1 ($1_{HAc} : 1_{NaAc}$)	CH_3CO_2H	H^+	$CH_3CO_2^-$
initial	0.01	0	0.01
@ equilibrium	0.01 - x	0 + x	0.01 + x

Self-dissociation of **Buffer #2**: Like with the above, the original concentration of the individual constituents (50mL 0.002M HAc and 50mL 0.02M NaAc) are altered to the new concentration of buffer 2 (0.001M HAc and 0.01NaAc); see formula 7.2;
these new concentrations (using formula 7.4, 7.5, and 7.6) yield the pH of the acetate-solution;

Buffer #2 ($1_{HAc} : 10_{NaAc}$)	CH_3CO_2H	H^+	$CH_3CO_2^-$
initial	0.001	0	0.01
@ equilibrium	0.001 - x	0 + x	0.01 + x

Formula 7.4:

$$K_{HAc} = \frac{[H^+]\cdot[Ac^-]}{[HAc]}$$

K_{HAc}, dissociation c. [mol/L]
[x], concentration [mol/L]

Formula 7.5: (quadratic equation: $0 = a\cdot x^2 + b\cdot x + c$) – see also appendix for software solution

$$_1x_2 = \frac{-b \pm \sqrt{b^2 - 4\cdot a\cdot c}}{2\cdot a}$$

a, b, c, constants [-]

Formula 7.6:

$$pH = -\log(x_1)$$

pH, hydrogen pot. [-]

Results: left the results of theoretical and right the measured pH-level (results indicated in grey)

	K_{HAc} [mol/L]	before mixing c_{HAc} [mol/L]	before mixing c_{NaAc} [mol/L]	buffing mixture c_{HAc} [mol/L]	buffing mixture c_{NaAc} [mol/L]	$[Ac^-] = [H^+] = x_2$ of quadr. EQ	pH theoret.	pH* measured
Buffer #1	1.8 E^{-5}	0.02	0.02	0.01	0.01	17.94E^{-6}	4.75	≈4.63
Buffer #2	1.8 E^{-5}	0.002	0.02	0.001	0.01	1.795E^{-6}	5.75	≈5.62

(*) see table of appendix

Remodulated expression of the quadratic EQ for:

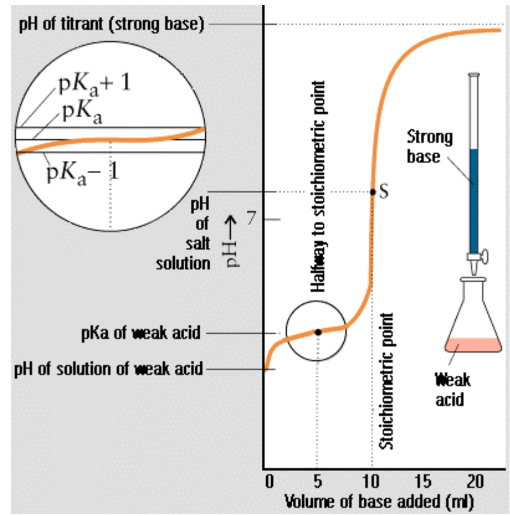
Buffer #1: $x^2 + x\cdot(K_{HAc} + 0.01) - 0.01\cdot K_{HAc} = 0$

Buffer #2: $x^2 + x\cdot(K_{HAc} + 0.01) - 0.001\cdot K_{HAc} = 0$

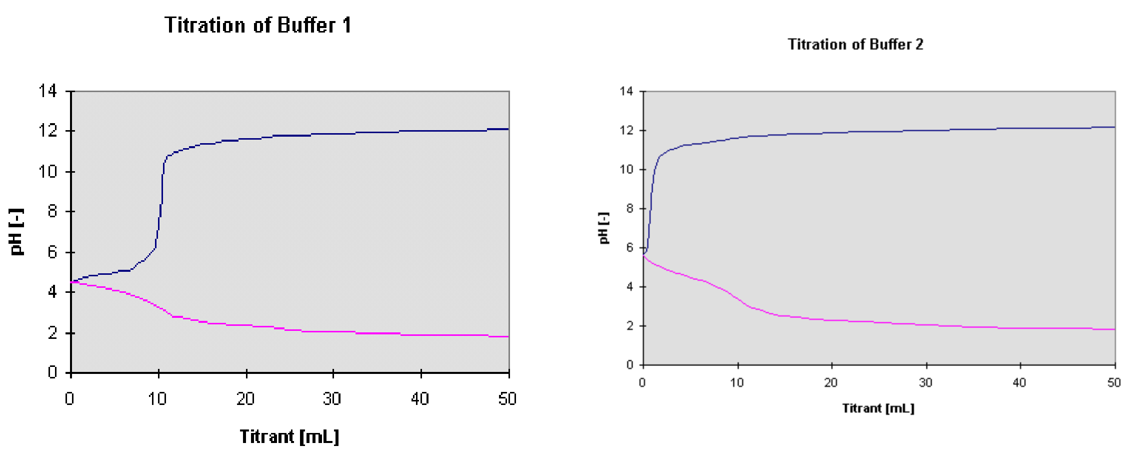
The theoretical results (using the mathematical procedure listed above) matches with the practically obtained pH (for both buffer 1 and 2).

The deviations are probably due to inaccuracies of the titrants used. Both the Reference Acid and the Reference Base are self-made titrants, which cannot be considered as “absolutely correct” values.

Evaluation: As indicated by the plots below, buffer #1 possesses far better buffing capacities than buffer #2.
The 10-fold increase of NaAc of buffer #2 yields a slightly better buffing characteristic when titrated with a strong acid as the improvements observed where in the order of just about one pH-unit compared when compared with buffer #1. Thus, the improvements are more or less insignificant.
When titrating buffer #2 with a strong acid, the buffer looses almost all of its properties. The over-supply of Na^+ ions from the NaAc, trims down the equilibrium reaction necessary in order to obtain a well working buffer; consequently, a sharp increase in pH is observed, even if only a few mL are added to the buffering solution.



Titration of a buffering solution (analyte) with a strong base



Titration results of the two buffer systems. Left chart represents buffer #1 (HAc/NAc in same amounts) while the right chart shows buffer # 2 (here the HAc is 10 times weaker than NAc); for amore detailed display of the graphs see appendix-tables.

A buffer with a high buffing capacity is obtained when the amount of analyte present is about 10% stronger (or even more) compared to the titrant; otherwise the buffer (analyte) gets used up quickly. This principle is valid for both the NaOH and the HCl used as the titrant with HAc/NaAc as the analyte.
The Henderson-Hasselbalch Equation shows that the corresponding pH range of the buffer is from:

$$pH = pK_a + \log\frac{[H^+]}{10\cdot[H^+]} = pK_a + \log\frac{1}{10} = pK_a - 1$$ for acid 10 times more abundant

$$pH = pK_a + \log\frac{10\cdot[HAc]}{[HAc]} = pK_a + \log\frac{10}{1} = pK_a + 1$$ for base 10 times more abundant

Experiment 8: - Determining the Reaction Enthalpy: Day 5: 6th of March 1998

8.1 Determination of the Neutralization Reaction-Enthalpy when mixing NaOH in contact with HCl:

Purpose: Measuring the enthalpy of neutralization in a reaction. The reference acid and reference base are used:

Base: NaOH(l) → $Na^+(aq) + OH^-(aq)$
Acid: HCl(l) → $H^+(aq) + Cl^-(aq)$
$H^+(aq) + OH^-(aq)$ → $H_2O(l)$ ($\Delta H = -55.6kJ$)
(see also experiment 1.2)

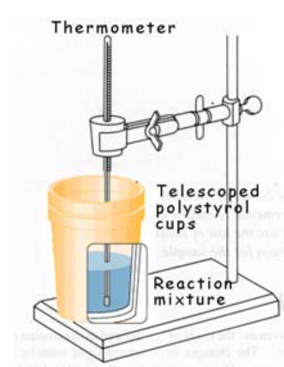
Being exergonic in nature, the enthalpy of reaction being results in a temperature increase that is significantly higher than ambient temperature.
Note: To make sure that the neutralization reaction is complete (all of the acid reacts with the base), it is far more efficient to weigh the reactants rather than to pipette the corresponding volume into the container.

material: 2 Stands w/ Burette-clamp
2 x 50ml burette
Pipette filler (Peleus) for 50mL volumetric pipette AS-class
2 x 100ml beaker
2 x labor-jacks
4 x Polyethylene or styrene cups
2 x Magnetic stirrer
2 x Hg-thermometers (0-30°C)
Digital single-pan balance
Protection glasses
Paper towels
Marker pen

chemicals:
≈110mL of Standardized Acid: HCl from exp.1; c = 0.980mol/L
≈110mL of Standardized Base: NaOH from exp.1; c = 0.988mol/L

Procedure: Determine the exact grams of NaOH (c = 0,980mol/l) and HCl (c = 0.988mol/l) needed to enable a complete neutralization reaction (formula 8.1 and 8.2);

- stack two polyethylene cups together, weigh them, and label each pair with their tara weights;
- while still on the balance, add a magnetic rod, place on digital flat balance and pipette the calculated mass of Standardized Acid (HCl) into the cup (see formula 8.2);
- using a 3rd polystyrene cup, record its tara weight and pipette the calculated mass of Standardized Base (NaOH) until the required mass is reached (minus tara of cup);
- place the cup containing the HCl and magnetic rod on the stirrer, turn on, and insert thermometer well below the upper liquid level; insert thermometer mounted on the stand into beaker containing the NaOH;
  Clue: slide the thermometer through a piece of cork and fasten it with a burette clamp onto the stand; to avoid that the magnetic rod will knock against the thermometer, use a lab-jack to lower and rise the acid containing cups;
  Note: avoid knocking with the magnetic rod against the tip of the thermometer; make sure that thermometer does not reach all the way to the bottom of the cups;
- recording of the Standardized Base temperature is not required as it is assumed that both the Acid and the Base do have the same temperature; otherwise allow both samples to acquire room-temperature (heat up with hand if necessary, or place the warmer on into a cold water bath - temperature difference should be <0.1K);
- record the temperature of the cup with the acid while still placed on the stirrer; quickly pour the base into the cup holding the acid and protocol the temperature change, during and after mixing in 20sec intervals at least for a minute, and in 1min intervals afterwards for further 3-4 mins);



Results and Evaluation: Execution of this experiment revealed a slight mismatching in pH of the Standardized Acid and Base; i.e. according to the tutors a faulty 3rd reference buffer must have been used during the calibration-procedure of the pH-meter; especially, since the shelf-life of the pH-10 reference buffer used in the calibration procedure of experiment 7, has already exceeded the imprinted due date. For approximation of density and concentration values, see data sheet (Appendix).

Mass of reactants to be weighed (to be determined individually for the acid and base):

Formula 8.1: ($n = c \cdot V$):

$$V_{reactant} = \frac{n_{reactant}}{c_{reactant}}$$

c, concentration	[mol/l]
V, volume	[L]

Formula 8.2: ($\rho = m/V$)

$$m_{reactant} = V_{reactant} \cdot \rho_{reactant}$$

n, molar amount	[mol]
m, mass	[g]
ρ, density	[g/L]

Formula 8.3: (for details see appendix-formula)

$$Q_{solution} = \frac{\Delta T \cdot m_{soluiton} \cdot 4.184}{n_{soluiton}}$$

T, temperature	[°K]
4.184	[J/(g·K)]
Q, energy	[kJ]

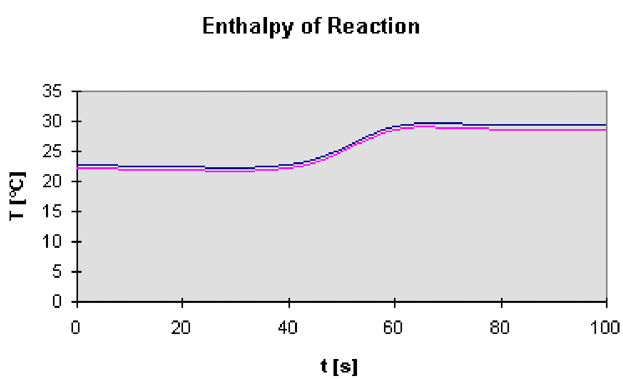
Data of mixture: $n_{NAOH} + n_{HCL} = n_{H2O} = n_{Na-} = n_{Cl-}$

	n [mol]	c [mol/L]	V [mL]	ρ* [g/L]	m [g]
acid-HCl	0.05	0.988	50.58	1016	51.41
base-NaOH	0.05	0.980	51.08	1040	53.08
∑	0.05				∑ 104.5

(*) density values have been approximated - see tables of appendix

Results of calculation (indicated in gray):

Trial	n [mol]	ΔT [°C]	Q [kJ/mol]
1st Trial	0.05	6.5	56.84
2nd Trial	0.05	6.35	55.53
averaged			56.184



The exergonic reaction enthalpy obtained is slightly above the reference value (**55.6kJ/mol**) but still within the tolerance window of ±3% (as requested by the tutors); for a more detailed plot see appendix-tables.

Experiment 9: - Conductivity of an Aqueous Solutions Day 7: 10th of March 1998

10.1 Serial Dilution:

Purpose: Sets of Serial Dilutions of three liquids (HCl, NaCl, and HAc,) have to be prepared, starting from highest ($10E^{-3}$) down to lowest ($100E^{-6}$ mol/L). Determine conductance of each dilution and plotted on paper.

Procedure: as the 0.098M Reference Acid is still available from experiment 1, we focus on the preparation of the **0.1M NaCl** solution (enough for all working groups):

- formula 9.1 yields the mass required to prepare this salty solution; weigh the calculated amount of mass into the 1L volumetric flask and fill with deionized water to 1L mark;

Preparation of **0.1M HAc** (enough for all groups):

- use again formula 9.1 to calculate the required mass and prepare this solution; weigh the calculated amount of mass, weigh it into the 1L volumetric flask and fill with deionized water to 1L mark;

1st **serial dilution** of HCl, NaCl, HAc (**10mM**):

- pipet 10mL from the 1L flask (0.1M HCl) into a 100mL volumetric flask and fill up w/ water;
- repeat for NaCl and HAc, using individual flasks;

2nd **serial dilution** of HCl, NaCl, HAc (**1mM**):

- pipet 10mL from the 100mL flask (10mM HCl) into a 100mL volumetric flask and fill up w/ H_2O;
- repeat for NaCl and HAc, using individual flasks;

3rd **serial dilution** of HCl, NaCl, HAc (**100µM**):

- pipet 10mL from 100mL flask (1mM HCl) into a 100mL volumetric flask and fill up w/ water;
- repeat for NaCl and HAc, using individual flasks;

Determine Conductivity of the 9 solutions, starting with the weakest concentration, rinse electrode when swapping from one type of solution to the next; i.e. change from HCL to NaCl, NaCl to HAc;

material: Stand w/ Burette-clamp
50mL burette
Pipette filler (Peleus) for 10mL volumetric pipette AS-class
6 x 100ml volumetric flask
2 x 1000mL volumetric flask
150mL beaker
Lab-jack
Magnetic stirrer
Electronic Conductometer w/ sensor
Digital single-pan balance
Protection glasses
Paper towels
Marker pen

chemicals: Deionized water
≈ 15mL of Reference Acid: HCl from exp.1; c = 0.0980mol/L
≈ 60mL of Reference Base: NaOH from exp.1; c = 0.0988mol/L
≈ 10g Acetic Acid glacial CH_3CO_2H (HAC, w = 100%)
≈ 10g solid Sodium Chloride NaCl (w = 99.5%)

Formula 9.1: (n = m/M)

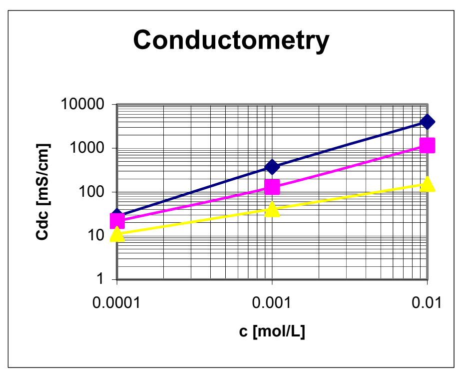
$m_{solute} = n_{solute} \cdot M_{solute}$	n, mole amount M, molar mass	[mol] [g/mol]

Obtaining a 0.1M solution (0.1M HCl already given):

	desired conc. [mol/L]	M [g/mol]	m [g]
NaCl	0.1	58.44	5.844
HAc	0.1	60.05	6.005

Results and Evaluation (measurements of conductance):

concentration		0.01 [mol/L]	0.001 [mol/L]	0.0001 [mol/L]
[µS/cm]	HCl	4010	374.2	22.92
	NaCl	1156	129.0	21.61
	HAc	153.2	40.81	11.03



Conductivity decreases with ion concentration in all of the three solutions (HCl blue, NACl pink, HAc yellow)

The solutes dissociate completely into their respective ionic constituents. In double-logarithmic scale and mainly due to hydration effects at low concentrations, conductometry reveals a linear relationship among the various molar concentrations. Although protons (H^+) are far more mobile than larger ions like Na^+, Cl^-, electrons are the most motile constituents within. Therefore, the meter detects charges only if electrons are allowed to move freely (saltatoric). Saltatoric motion can only take place when "hydration jackets" are overcome.

10.2 Determining the Equivalence Point of a monoprotic acid:

Purpose: Calculate the concentration of a monoprotic acid sample obtained from the tutors (acid in which only one proton dissociates from the molecule). The concentration can easily be found when monitoring the conductivity of the monoprotic acid analyte while titrating it with the Reference Base of experiment 1 (0.098M NaOH).

Procedure: Rinse all utensils with deionized water, mount titration stand with magnetic stirrer and obtain few millilitres of the unknown monoprotic acid sample using a 100mL volumetric flask;

- fill up a 100mL volumetric flask containing the unknown acid with deionized water;
- for the titration, rinse the 50mL burette with the titrant (0.1M Reference Base), mount burette along with conductivity sensor onto the stand, and pour the titrant it up to zero-mark;
- extract 25mL of this flask and pipet it into a 150mL beaker, add another 50mL of deionized water, add magnetic rod, place on magnetic stirrer, and start titration;
  Clue: record the conductivity after every exactly 1mL of titrant used;
- draw a chart and interpolate cross-point of both the descending and ascending trend lines (theoretical volume of titrant NaOH);
- by using formula 6.3 and 6.4, calculate the concentration and exact mass percentage of the monoprotic (analyte);

Results and Evaluation: Conductivity measurements of the titrated analyte yielded the following results (for data, see appendix-table). By graphically interpolating the descending and ascending branches of the graph, one obtains with satisfying accuracy the volume of titrant used to reach complete neutralization of the monoprotic acid. (using formula 9.2).

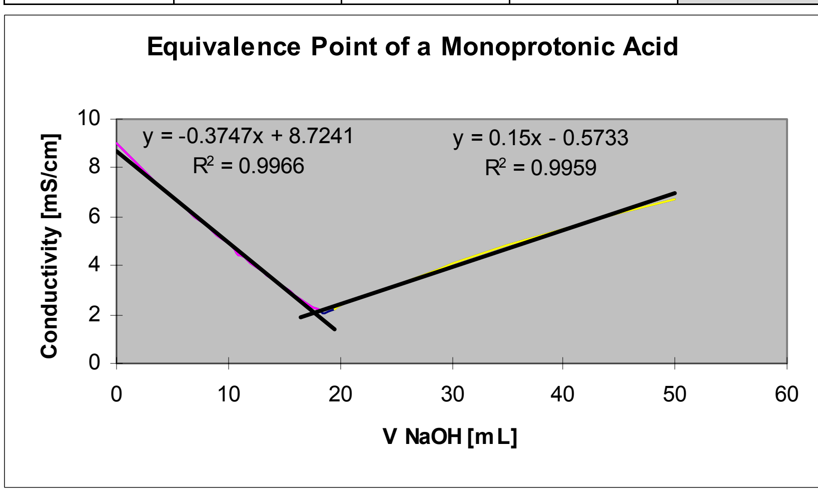
Formula 9.2: ($c_1 \cdot V_1 = c_2 \cdot V_2$)

$$c_{acid} = \frac{c_{NaOH} \cdot V_{NaOH}}{V_{acid}}$$

c, concentration [mol/l]
V, volume [L]

Results of Titration (result indicated in grey):

Evaluation	Titrant@Equivalence V_{NaoH} [mL]	Titrant@Equivalence c_{NaoH} [mol/L]]	Analyte@Equivalence V_{acid} [mL]	Analyte@Equivalence c_{acid} [mol/L]
graphic	18.25	0.098	25	71.54·E-3
mathematic	17.72	0.098	25	69.46·E-3



Measuring Conductivity while titrating

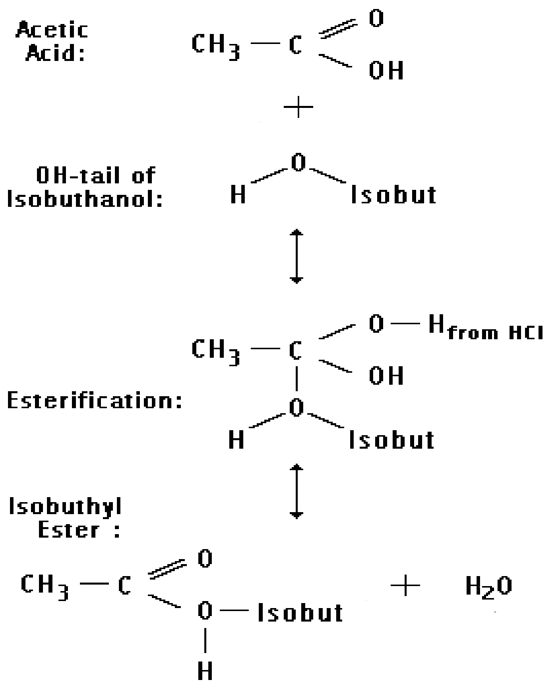
Based on the graphical evaluation, the initial H^+ concentration of the monoprotic test-acid must have been 71.54mM (approx pH 1.1). Based on the mathematical evaluation, the monoprotic test-acid must have been 69.46mM. Verification by the tutors conformed that both results are within the margin of error (±3%); see appendix-tables for a more detailed plot.

Experiment 10.

Esterification and Determination of Equilibrium Constant: Day 6: 9th of March 1998

10.1 Esterification with Reflux technique:

Purpose: Esterification of ethanol or isobuthanol (IBOH) can be done with Acetic Acid and HCl used as a catalyst to generate ethyl acetate or isobutyl acetate. Using the dehydrated EtOH from experiment 3, the product of esterification will yield a glue-like smell; using IBOH, the product would have a banana-like aroma. Our working group opted for IB rather then EtOH:



Hydrochloric acid is needed prevent dissociation and to render the centremost C-atom of the HAc more positive. The dissociated proton (H^+) from the HCl gets hold of the double-bonded O-atom of the HAc's carboxyl group, and thus weakens the shielding charge-coupled cloud around the centremost C-atom. This enables the OH-tail of the IBOH to launch its nucleo-philic attack on the centremost C-atom of the HAc molecule. The HAc's carboxyl group in turn draws back the charge-coupled cloud towards the newly developing CO-bond of the attaching IBOH to form isobuthyl-ester (IBE)

material: 2 Stands w/ Burette-clamp
- 50ml burette
- Pipette filler (Peleus)
  - 1mL volumet. pipette AS-class
  - 2mL volumet. pipette AS-class
  - 10mL volumet. pipette AS-class
  - 25mL volumet. pipette AS-class
  - 500mL 3-arm round-bottom flask with stand & electrically powered heating mantle
- 50ml volumetric flask
- 15mL beaker
- 300mL beaker
- 1L beaker w/ some shredded ice
- 300mL wide-mouthed Erlenmeyer
- Lab-jack
- Magnetic stirrer
- Hg-thermometers (50-150°C)
- Water cooled dockable Reflux-Dimroth condenser
- Dockable drier unit for condenser filled with $CaCl_2$
- Stopcock grease
- Digital flat-pan balance
- Protection glasses
- Paper towels & Marker pen

chemicals: Deionized water
- 3 boiling chip granules
- 25mL HCl (w = 32%)
- ≈60mL Acetic Acid (w = 100%)
- ≈100mL $(CH_3)_2CHCH_2OH$ Isobuthanol (IBOH) or EtOH from exp. 3
- ≈200mL of Reference Base: NaOH from exp. 1; c = 0.0988mol/L
- few drops of phenolphthalein -indicator (PP)

Procedure: Assemble reflux-apparatus on stand w/ clamps (incl. $CaCl_2$ charged drier), magnetic stirrer, round-bottom flask with electric heater mantle; **Note**: do not forget to seal joints with grease;
Diluting the concentrated HCl (w = 32% ≈10M, see table in appendix):
- pipet 25mL from the concentrated HCl into a 50mL volumetric flask and add deionized water until to the marked upper limit to obtain a diluted HCl with a mass percentage of 16% (≈5M);

Preparation of solution to favour the above reaction (use 3rd vacant side arm of rounded flask)
- weigh exactly 70g of IBOH, 60g of HAc (or actual weight used) and pour into rounded flask;
- add exactly 5mL of diluted HCL (w = 16%) and place 3 pieces of boiling chips into the flask;
- pop sidearm with glass stopper, and reflux (boil for ≈2 hours); allow to cool off; in the meantime proceed with procedure 10.2 (titration).

10.2 Titration of diluted HCL and Reaction sample:

Procedure: Before being able to determine the Equilibrium Constant (K_C) of the reaction between IBOH and IBE, the molar concentration of the HAc and the HCl must be known. As with experiment 1, set up the titration stand with magnetic stirrer and lab jack; clean 50mL burette with deionized water and:

- rinse it with the titrant (0.098M NaOH Reference Base), close stopcock and fill it up to zero-mark;

Titration of HCl - preliminary verification of diluted HCl (w = 16%):

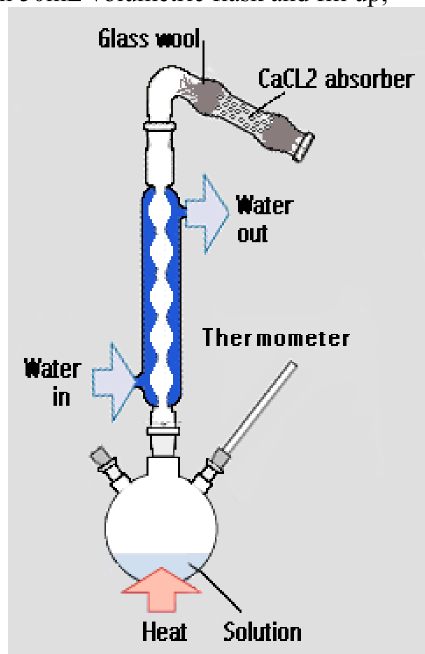
- further stretch the HCl (to 1:10, w = 1.6%); i.e. pipet 5mL in 50mL volumetric flask and fill up;
- pipet 2mL of the analyte (diluted HCl) into a 300mL wide-mouthed Erlenmeyer flask, add approx. 100mL of deionized water and a drop of PP-indicator;
- titrate anylate and determine concentration by using formula 10.1;
- repeat titration two to three times and average results;

Titration of IBE sample – determination of the remaining proton-activity (H^+) of HAc & HCl (after two hours):

- use same titrant as above (0.098M NaOH);
- extract 10mL of IBE and pipet into a 15mL beaker; **Note**: to avoid cracking of the beaker, make sure that the isobuthyl-ester has cooled off;
- place the 300mL wide-mouthed Erlenmeyer flask into the 1000mL beaker filled with shredded ice;

1. pipet 1mL of the 10mL IBE-solution along with 50mL of deionized water into the Erlenmeyer flask, add a drop of PP-indicator and start titration;
2. stop titration when color change takes place, and record consumed volume of NaOH-titrant;

- repeat titration steps 1 & 2 to obtain a total of three separate results and average volume of titrant used.



Reflux apparatus used for the esterification of IBOH with HAc

Results and Evaluation: According to data sheets, the molar concentration of the diluted HCl (w = 16%) should be approx. 4.65 mol/L.

Formula 10.1: ($c_1 \cdot V_1 = c_2 \cdot V_2$)

$$V_{conc.} = \frac{c_{diluted} \cdot V_{diluted}}{c_{concentrated}}$$

c, concentration [mol/l]
V, volume [L]

Results of Titration (indicated in gray) HCl titration IBE sample

Titration	Titrant V_{NaOH} [mL]	Titrant c_{NaOH} [mol/L]	Analyte V_{HCl} [mL]	Analyte $c_{HCl\,(1.6\%)}$ [mol/L]	Analyte $c_{HCl\,(16\%)}$ [mol/L]		IBE sample Titrant V_{NaOH} [mL]
1st	10.70						20.2
2nd	10.65						20.5
3rd	10.66						20.2
averaged	10.67	0.0980	2	0.523	5.23		20.3

The preliminary test-titration (stretched HCl sample, w = 1.6%) revealed a concentration of 0.523mol/L (which corresponds to 5.23M of the diluted HCl sample), a value well inline with those recorded by the other working groups.

To reach the equivalence point, the titration of the IBE sample used up an average volume of 20.3mL of NaOH titrant. This amount along with the formulas listed on the next page is used to calculate the residual molar amount of IB left (within the approx. 150mL) after esterification to IBE has taken place.

10.3 Determining of Equilibrium Constant (K_C) of this reaction:

According to the sketch shown o page 24, the reaction can be summarized as follows (with IBOH standing for Isobutanol; and IBE indicating the Isobuthyl-Ester):

$$1\ HAc + 1\ IBOH \leftrightarrow 1\ IBE + 1\ H_2O$$

Thus, the overall molar amounts are given by:

The amount of HAc left over at the end of the reaction…….. $n_{HAc@end} = n_{HAc@end} + n_{HCl@end} - n_{HCl@start}$

The amount of IBE yielded, depend on the amount of HAc consumed during the reaction: …………..………….. $n_{IBE@end} = n_{HAc@start} - n_{HAc@end}$

For each IBE formed, 1 H_2O molecule hydrolyzes; therefore, additional H_2O must be derived from the diluted HCl: $n_{H_2O} = n_{HAc@start} - n_{HAc@end} + n_{H_2O\ from\ dil.HCl}$

The amount of IBOH left after esterification to IBE depends on the amount of HAc left …………………………..….. $n_{IBOH@end} = n_{IBOH@start} - n_{HIBE@end}$

Results and Evaluation: According to the molar relations stated above, we need to determine the molar amounts of: $n_{IB\text{-}at\ start}$, $n_{Hac\ at\ start}$, $n_{H2O\ at\ start}$, $n_{Hac\ at\ start}$, $n_{Hac\ at\ end}$

Formula 10.2: (ρ = m/V)	$m_{solutn} = \rho_{solutn} \cdot V_{solutn}$	ρ, density m, mass	[g/L] [g]
Formula 10.3:	$n_{solute} = m_{solute} / M_{solute}$	M, molar mass	[g/mol]
Formula 10.4: ($c_1 \cdot V_1 = c_2 \cdot V_2$)	$V_{conc.} = \frac{c_{diluted} \cdot V_{diluted}}{c_{concentrated}}$	c, concentration V, volume	[mol/L] [L]
Formula 10.5:	$n_{solution} = c_{solution} \cdot V_{solution}$	n, molar amount	[mol]
Formula 10.6: (see appendix-formula)	$K_C = \frac{n_{IBE@end} \cdot n_{H_2O@end}}{n_{HAc@end} \cdot n_{IBOH@end}}$		

Determining *n* and *V* of IBOH and HAc at start of the reaction (using first formula 10.3, then 10. 2):

Isobutlhanol (IBOH)					Acetic Acid (HAc)				
m [g]	M [g/mol]	n [mol]	ρ[1] [g/L]	V [mL]	g [g]	M [g/mol]	n [mol]	ρ[1] [g/L]	V [mL]
70.05	74.12	0.945	800	87.56	60	60.05	0.992	1050	57.14

Thus, the overall volume in the reaction flask calculates as: $V_{total} = V_{HCl} + V_{HAc} + V_{IBOH}$ = **144.8mL**

Determining *n* of the diluted 5mL HCl added to the reaction flask (using formula 10.2 & 3):

$HCl_{w=16\%}$			84% water content			16% acis content				Aliquot in 1mL		
ρ[1] [g/L]	V [mL]	m_{acid} [g]	$_{84}m_{H2O}$ [g]	M_{H2O} [g/mol]	$_{84}n_{H2O}$ [mol]	$_{16}m_{HCl}$ [g]	M_{HCl} [g/mol]	$_{16}n_{HCl}$ [mol]		volumetric factor		n_{HCl} [mol]
1078	5	5.390	4.527	18.02	0.251	0.862	36.46	23.65 E-	/	144.8	=	163.4E-6

Molar extrapolation the titration volume (1mL) to that in reaction flask (using formula 10.5) and the relationship: $n_{HAc@end} = n_{NaOH} - n_{HCl}$; to rounded reaction flask:

Titrant (1M NaOH)				Analyte (extracted 1mL sample)				Aliquot in 145mL		
V [mL]	c [mol/L]	$n_{NaOH} = n_{HAc+HCl}$ [mol]		$_{16}n_{HCl}$ [mol]		$_{16}n_{HAc}$ [mol]		volumetric factor		$n_{HAc@end}$ [mol]
20.3	0.098	1.989 E-3	-	158.0E-6	=	1.83 E-3	*	144.8	=	0.264

Values determined by the molar relationships given at the top of this page (to be used in formula 10.6):

$n_{HAc@end}$ [mol]	$n_{IBE@end}$ [mol]	$n_{H2O@end}$ [mol]	$n_{IBOH@\ end}$ [mol]	K_C [mo/Ll]
0.264	0.735	0.986	0.210	13.04

([1]) density values approximated from data sheet – see table of appendix

Although K_C can only verified theoretically, the undeniable smell of "banana" indicated that the reaction took place.

Experiment 11. Nitrate determination w/ Spectrometer: Day 8: 11th of March 1998

11.1 Calibration of spectrometer:

Purpose: In order to determine the nitrate (NO_3^-) concentration of three unknown water samples, it is necessary to calibrate the photo-spectrometer with reference solutions of known nitrate concentration (5 gradually increasing nitrate concentration). To assign the nitrate light-absorptive properties it is necessary to "stain" the dissolved NO_3^-.

material: Pipette filler (Peleus)
2mL volumet. pipette AS-class
10mL volumet. pipette AS-class
20mL measur. pipette AS-class
500mL volumetric flask
250mL volumetric flask
10 x 100mL volumetric flask
8 x 80mL beakers
50mL beaker
2 hot-plates
Spectrophotometer
Digital single-pan balance
Oven (max. 200°C)
Protection glasses
Paper towels
Marker pen

chemicals: Deionized water (nitrate free!)
24 boiling chip granules
0.5g Sodium Salicylic Acid $C_7H_5O_3Na$
1.37g Sod. Nitrate $NaNO_3$ (w = 99%)
15g pure Potassium Sodium Tatrate-tetra hydrate $C_4H_4O_6KNa{\cdot}4H_2O$
100g Sodium Hydroxide pellets NaOH (w = 99%)
≈30mL Sulf. Acid H_2SO_4 (w = 96%)
≈100mL $(CH_3)_2CHCH_2OH$
100mL tap-water sample
100mL water sample - tutors
100mL water sample - dr.Malissa

Procedure: preparation of the nitrate solutions;

Solution-1 (used as a staining reagent in all samples):

- mix 100g of NaOH pellets and 15g K-Na Tatrat in a 250mL volumetric flask and fill up to the mark with deionized water; **Note**: Execute this step in cold water bath (reaction is strongly exothermic).

Solution-2 (used to prepare unknown samples):

- put 0.5g Na-Salicylic Acid into a 100mL vol. flask; and fill up to the mark w/ deionized water.

Solution-3: Reference Dilution Series used to calibrate photo-spectrometer (matrix & NO_3^- mixture);

- weigh 1.370g of $NaNO_3$, place in 100mL vol. flask and fill up with deionized water (V_{100});
- pipet 5mL (V_{pipet}) of this solution into a 500mL vol. flask (V_{500}), fill up water (formula 11.1);
- prepare 5 beakers (80mL) and extract the following volumes of diluted reference solution: 15mL into 1st beaker; 10mL into 2nd; 5mL into 3rd; 2mL into 4th; none into 5th beaker (matrix only);
- add ≈50mL of deionized water, 3 boiling chips, and 2mL of solution-2 to each beaker.

Preparation of sample solutions to be analysed:

- pipet **exactly** 50mL of: tap-water sample into 6th beaker, 50mL tutor's water sample into 7th and 50mL dr.Malissa's water sample into the 8th beaker
- add 3 boiling chips, and 2mL of **solution-2** to each beaker;

Treatment of Reference Dilution Series (5 varying concentrations) and the 3 unknown samples:

- place all eight beakers onto a hot plate and simmer until all the water has evaporated (≈1hour); **Note**: do not burn residues; don't boil too long; too much solute might get lost (falsifying results);
- dry beakers in 100-110°C warm oven until residual humidity is gone;
- once beakers have cooled down, add 2mL of H_2SO_4(w = 96%) to each beaker, wait until residues have dissolved completely, wait another10mins; then add 15mL of deionized water to each beaker; **Note**: do use protection glasses! If this order is **not** followed, mixture might react violently!
- for the final staining reaction add 15mL of **solution-1** to each beaker (turns yellowish); allow to cool down and shake well;
- transfer content of each beaker into separate 100mL vol.-flasks; fill up w/ deion. water, and shake.

Formula 11.1: (for details see appendix-formula)

$$\beta_{NO3^-} = \frac{m_{NaNO_3} \cdot M_{NO3^-} \cdot V_{pipeted}}{M_{NaNO_3} \cdot V_{100} \cdot V_{500}}$$

β, mass concentration [g/L]
V, volume [L]

Results of the Reference Dilution Series of Solution-3 (results indicated in grey):

Solution-$3_{dil.}$	Beaker #1	Beaker #2	Beaker #3	Beaker #4	Beaker #5	
β_{NO3^-} [mg/L]	99.95	29.98	19.98	9.995	3.998	0.0

11.2 Spectrometric Analysis:

Purpose: The light-absorbing characteristics of each sample correlates with the nitrate content in each of the stained samples. By using the 0mg/L sample, the photo-spectrophotometer is first set to zero. In a 2nd step the Reference Dilution Series is used to "calibrate" the slope of the detecting sensor of the instrument. Measurements of the unknown samples can then be executed.

Procedure: Calibrating photo-spectrometer to zero:

- use the appropriate cuvettes, and fill both reference and sample cuvette with the 0mg/L sample (Ref.Dil.Series - sample 5); place into scan compartment and adjust tuning knob to 0;
  Note: Do not touch cuvet at scan-window; working with yellowish probes, the scanning wavelength should be set to 420nm (this is the wavelength at which maximum absorption of the nitrate occurs and is used to obtain max. photometric sensitivity for various nitrate concentrations);

1. remove sample cuvet from machine, flush cuvet twice and fill with next in line (Ref.Dil.Series - sample 4)
2. record light- extinction as given by the detector;
3. repeat steps 1 and 2 with all other Ref.Dil.Series samples as well as with the 3 unknown test samples;
4. plot a chart by using the Ref.Dil.Series data set (5 different data points);
5. determine nitrate concentration of unknown sample solutions.

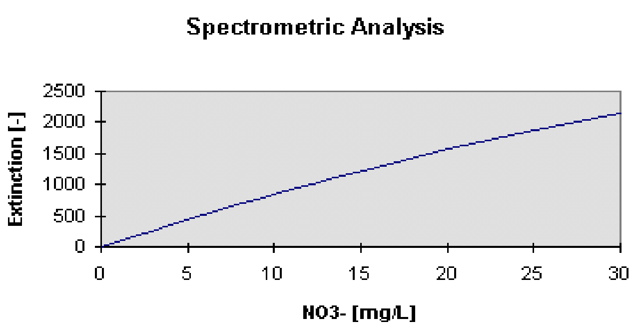
The side of this spectrophotometer has been opened so that we can see the spectrum generated by the diffraction grating inside. The grating is rotated so that the light of the desired wavelength falls on the sample.

Results and Evaluation: While swapping prepared solution from 80mL beaker into 100mL volumetric flask, some of the Reference Dilution Series sample 4 has been spilled; therefore, the entire preparation of sample 4 (2mg/L) has been repeated....☹.
Evaluation with the freshly prepared Reference Dilution Series sample 4 confirmed the already linear relationship of the dilution series. Using the slope of the graph to match the extinction coefficient with the calibration curve of the Dilution series yielded the NO_3^- content of the unknown samples. The results obtained from the photo-spectrometric analysis matched perfectly with the cross-reference data of the tutors.

Results spectrometric analysis (indicated in grey):

	Calibration samples (matrix & NO_3^-)					Test samples		
	$1_{Calibr.}$	$2_{Calibr.}$	$3_{Calibr.}$	$4_{Calibr.}$	$5_{Calibr.}$	$6_{Malissa}$	7_{Tutor}	7_{Tap}
$S\text{-}Meter_{Reading}$	2.137	1.630	0.783	0.342	0	1.039	0.817	0.522
NO_3^- [mg/L]	29.98	19.98	9.995	3.998	0.0	13.75	10	6.5

see also appendix-tables for a more detailed chart



Calibration series and unknown NO_3^- samples

Exp. 12: Hardness of Water (Alkaline Earth Metals): Day 8: 11th of March 1998

Purpose: The purpose of this experiment is to determine the hardness of a tap-water sample and an unknown probe provided by the tutors.

Ca^{2+}/Mg^{2+}-ions are bounded to the EDTA when brought together. To favour this reaction, a high basic medium is needed (ammonium solution); only then the alkaline earth metals (in a process called coating) readily binds to the EDTA complex (dynamic equilibrium). As these metals have a low stability constant, a high concentration of the EDTA titrant at a high pH is required (>10). A bit of ECBT indicator is added to reveal the presence of any dissociated Ca^{2+}/Mg^{2+}-ions in solution. The colour change from red to blue is brought about by the strong interaction of the EDTA (EDTA is capable of breaking the less stable ECBT metal-complex).

$CaCO_3 \leftrightarrow Ca^{2+} + 2HCO_3^-$

material: Stand w/ buret clamps &
- 10mL burette
- Pipette filler (Peleus)
- 1mL volumet. pipette AS-class
- 10mL volumet. pipette AS-class
- 25mL volumet. pipette AS-class
- 100mL volumetric flask
- 2 x 80mL beakers
- 300mL wide-mouthed Erlenm. flask
- Lab-jack
- Magnetic stirrer
- Digital flat-pan balance
- Protection glasses
- Paper towels
- pH-Indicator paper
- Marker pen

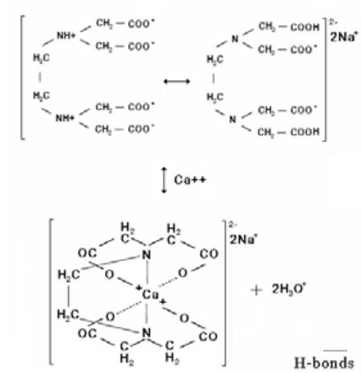
chemicals: Deionized water
- 10mL Titriplex III for metal titration Na_2-EDTA-$2H_2O$ (c = 0.1M)
- ≈40mL Ammonia Solution NH_3 (w = 25%)
- 5.35g Ammonium Chloride NH_4Cl (w = 99%)
- a pinch of ErioChrome Black T-Me indicat. $C_{20}H_{12}N_3NaO_7S$ (ECBT)
- ≈60mL tap-water sample
- ≈60mL water sample from tutors

Procedure: Rinse all utensils with deionized water and clamp 10mL burette onto the stand.

Preparation of titrant (0.01mol/L EDTA):
- pipet 10mL of EDTA into a 100mL volumetric flask and dilute with deionized water;
- rinse buret w/ diluted EDTA and fill up;

Preparation of buffer (stabilizes pH at 10 to 11)
- weigh 5.35g NH_4Cl into the 100mL volumetric flask and dissolve with some deionized water;
- add 35mL NH_3, fill up with deionized water and shake;

Titration (confirm pH with indicator paper):
- pipet 25mL of tap-water into the 300mL wide-mouthed Erlenmeyer flask, add 1mL of buffer, and add a pinch of ECBT indicator to the analyte (solution turns red);
- titrate, and record volume of used titrant (change to blue),
- repeat titration to obtain at least two extra readings;
- repeat titration with sample obtained from tutors;
- convert the data to mass-equivalents (formula 12.1) of [mg] CaO and calculate hardness of water sample (formula 12.2)



EDTA-chelate of alkaline earth metals

Results and Evaluation of Titration:

Formula 12.1: (for details see appendix-formula)

$$m_{CaO} = \frac{c_{EDTA\ titrant} \cdot V_{EDTA\ titrant} \cdot 56.1}{V_{H_2O\ Sample}}$$

c, concentration [mol/L]
V, volume [L]

Formula 12.2: (for details see appendix-formula)

$$dH_{H_2O} = m_{CaO} \cdot 100$$

m, mass [g]
dH, hardness [°]

Results of titration (indicated in grey):

Titration	tap-water sample					tutor's water sample				
	$V_{Titrant}$ [mL]	$c_{Titrant}$ [mol/L]	V_{H2O} [mL]	m_{Cao} [mg]	dH [°]	$V_{Titrant}$ [mL]	$c_{Titrant}$ [mol/L]	V_{H2O} [mL]	m_{Cao} [mg]	dH [°]
1st	4.85					9.2				
2nd	4.62					9.2				
averaged	4.735	0.01	25	106.3	10.63	9.2	0.01	25	206.5	20.65

Experiment 13: Sulfate concentration Day 9: 12th of March 1998

13.1. Chromatography using an Ion exchanger:

Purpose: Sulfate compounds can be stripped from their cationic counterparts by passing them through a ion-exchanger. Cations like Cu^{2+} are held back and are swapped against H_3O^+ ions. Due to electrostatic repulsion, anions like SO_4^{2-} can pass unhindered. To make this happen, the cation-exchanging pellets must be charged; e.g. with H^+. Charging is achieved by rinsing the column with a monoprotic acid (e.g. HCl).

Charging column: $2HCl \rightarrow column \rightarrow CuCl_2 + 2H^+$
Ion Exchange: $CuSO_4 + 2H^+ \rightarrow column \rightarrow Cu^{2+} + H_2SO_4$

material: Stand w/ buret clamps & 10mL burette
- Ion-exchanger column filled with polystyrol-resin pellets
- Pipette filler (Peleus)
  - 5mL measur. Pipette AS-class
  - 5mL volumet. Pipette AS-class
  - 50mL volumet. Pipette AS-class
- 100mL graduated cylinder
- 5 x 100mL volumetric flask
- 250mL volumetric flask
- 500mL volumetric flask
- 1L volumetric flask
- 3 x 300mL wide-mouthed Erlenmeyer
- Lab-jack
- Magnetic stirrer
- Digital single-pan balance
- Protection glasses
- PH indicator paper
- Paper towels
- Marker pen

chemicals: 2.5L Distilled water (Micropore)
- 50mL Na_2-EDTA-$2H_2O$ (c = 0.1M) Titriplex III for metal titration
- 4.886g Barium Chloride $BaCl_2 \cdot 2H_2O$ (w = 99%)
- ≈5g Hydroxide Ammonium Chloride $HONH_3Cl$ (w = 99%)
- 0.5g Dipotassium-Magnesium EDTA $C_{10}H_{12}K_2MgN_2O_8 \cdot 2H_2O$ (w=99%)
- 3.5g Amon. Chloride NH_4Cl (w=99%)
- a pinch of ErioChrome Black T-Me $C_{20}H_{12}N_3NaO_7S$ (ECBT)
- ≈20mL Hydrochl. Acid HCl (w= 32%)
- 100mL Amon. Solution NH_3 (w=25%)
- ≈100mL EtOH from exp. 3 C_2H_5OH (w = 100%)
- $CuSO_4$ sample in 100mL Erlenm.

Procedure: Preparation of the 2M HCl:
- Pipet the calculated volume of concentrated HCL into a 100mL volumetric flask (formula 13.1) and fill up with distilled water;

Charging the ion-exchanger with protons:
- mount the column onto stand and rinse with approx. 100mL *distilled* water;
  Note: No air should be trapped within the column; keep liquid level above granule level.
- pour the 2M HCl-solution into the column of the exchanger (adjust Stopcock to a drip rate ≤4 drops a second);
- rinse charged column with *distilled* water (≈ 100mL), use pH-paper to monitor the pH of the outflow;
  Note: keep liquid level above granulate level; pH at outlet should match that at top of exchanger

Executing the ion exchanger routine:
- obtain the a water-sample from the tutors (few mL pipetted in 100mL volumetric flask); fill up with ultra pure *distilled* water and shake well (V_{Probe});
- pipet 50mL ($V_{ion\text{-}Exchange}$) into the exchanger and collect effluent in a 250mL volumetric flask; adjust Stopcock valve to obtain a drainage rate of 4mL/min (total effluent time approx.: 12mins);
- flush exchanger with 75mL *distilled* water; add effluent to the previous probe in flask; shake well;
- fill flasks with *distilled* water until the 250mL ($V_{Analyte}$) mark and proceed with 13.2.

Formula 13.1: ($c_1 \cdot V_1 = c_2 \cdot V_2$)

$$V_{conc.} = \frac{c_{diluted} \cdot V_{diluted}}{c_{concentrated}}$$

c, concentration [mol/l]
V, volume [L]

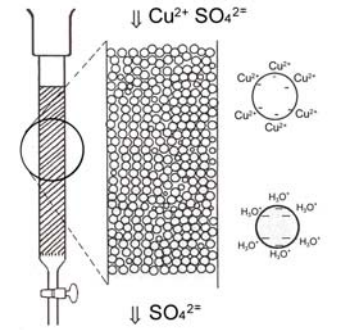
Results of Regeneration (indicated in grey):

	$c_{diluted}$ [mol/L]	$V_{diluted}$ [mL]	$c_{concentrated}$ [mol/L]	$V_{concentrated}$ [mL]
HCl	2	100	approx. 10*	20

(*) concentration obtained from data sheet, see appendix

13.2. Titration:

Purpose: The acidic effluent (now H_2SO_4) collected in the Erlenmeyer flasks, is mixed with a salt of an alkaline earth metal (e.g. $BaCl_2$) of known concentration. In the presence of dissociated sulfate ions ($SO_4^{=}$), Barium ions (Ba^{2+}) form a non-dissolving precipitate ($BaSO_4$). The amount of Ba-salt used is such as to leave some of the Barium (Ba^{2+}) in solution once all sulfate ions have been bound to the precipitate. The remaining Ba^{2+} ions can be titrated with EDTA. The amount of EDTA consumed is equivalent to the dissociated Ba^{2+} ions in solution. Knowing the initial amounts of Ba-salt added and the amount of precipitate following the reaction, the Barium in the solution is inversely proportional to the SO_4^{2-} content in the precipitate. An ECBT-indicator is used to monitor the colour change from red to blue when all of the aqueous Barium is in solution is trapped in the EDTA-Ba-complex:



The ion-exchange mechanism occurring on the surface of the polystyrol-resin pellets

$Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)$
$Na_2\text{-}EDTA(l) \rightarrow 2Na^{+}(aq) + EDTA^{2-}(aq)$
$Ba^{2+}(aq) + EDTA^{2-}(aq) \rightarrow Ba\text{-}EDTA\ (aq)$
once no Ba^{2+} is left in solution, additional Na_2-EDTA results in a colour change of the indicator

Procedure: Rinse all utensils with distilled water and clamp 10mL burette onto the stand.

Preparation of titrant (0.01M Na_2-EDTA; enough for one working group):

- pipet 10mL into a 100mL volumetric flask, fill up with *distilled* water, and shake well;

Preparation of buffer solution (stabilizes pH at 11; enough for all working groups):

$NH_4Cl \rightarrow NH_4^{+} + Cl^{-} \quad NH_4^{+} \rightarrow NH_3 + H^{+}$

- weigh 0.5g of K_2Mg-EDTA, 3.5g NH_4Cl, and 90mL NH_3 into a 100mL volumetric flask, fill up with *distilled* water and shake well;

Preparation of 0.02M $BaCl_2$ solution (enough for all working groups): $BaCl_2 \rightarrow Ba^{2+} + 2Cl^{-}$

- weigh 4.886g $BaCl_2$ into a 1L volumetric flask, fill up with *distilled* water, and shake well;

Preparation of Indicator solution (enough for all working groups):

- weigh 0.5g ECBT indicator and 4.5g $HONH_3Cl$ with 100mL EtOH in another volumetric flask;

Titration: pipet 50mL ($V_{dil.Analyte}$) of the water sample from the 250mL flask ($V_{Analyte}$) into each of the individual 300mL Erlenmeyer flasks;

- add 5mL of 0.02M $BaCl_2$ solution to each flask, boil them for 5mins, keep simmering for further 15mins, and allow content in flasks to cool down for another 90mins;
- pipet 4mL of buffer, and 1 or 2 drops of indicator (solution turns dark red) to each flask;
- rinse buret with titrant (Na_2-EDTA) and fill up to zero mark;
- titrate each flask individually, and determine the concentration of sulfate ions (formula 13.2).

Results and Evaluation: the results obtained were found to be within the 3% margin as requested by the tutors (4.6µmol/L SO_4^{2-}); converting

Formula 13.2: (for details see appendix-formula)

$$c_{SO_4^{=}} = \frac{\left(c_{Ba^{2+}} \cdot V_{Ba^{2+}} - c_{titrant} \cdot V_{titrant}\right) \cdot V_{analyte}}{V_{dil.analyte} \cdot V_{ion\text{-}exchanger}}$$

c, concentration	[mol/L]
V, volume	[L]
m, mass	[g]
M, molar mass	[g/mol]

$$\beta_{SO_4^{=}} = c_{SO_4^{=}} \cdot M_{SO_4^{=}}$$

Results of titration end dilution (indicated in grey):

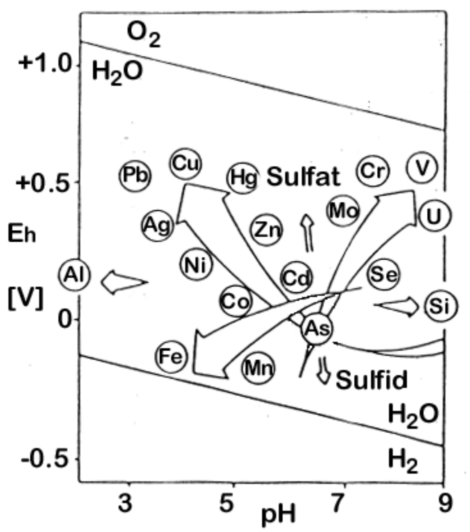
Titration	$BaCl_2$		Titrant		Analyte		Ion-Exchanger		SO_4^{2-}		
	$V_{Ba^{++}}$ [mL]	$c_{Ba^{++}}$ [mol/L]	V_{EDTA} [mL]	c_{EDTA} [mol/L]	$V_{Analyte}$ [mL]	$V_{dil.Anal.}$ [mL]	$V_{ion\text{-}Exch.}$ [mL]	V_{Probe} [mL]	c_{final} [mol/L]	$M_{SO4=}$ [g/mol]	β_{final} [g/L]
1st			5.43								
2nd			5.48								
3rd			5.49								
averaged	5	0.02	5.437	0.01	250	50	50	100	.563E-3	96.06	0.438

Experiment 14: Qualitative Analysis Day 10: 13^{th} of March 1998

Purpose: Analysing a three different sample in search for the dissolved ionic heavy metal constituents. Simply exposing them to high concentrated HCl triggers a precipitation reaction of the dissolved Me-ions.

material: Pipette filler (Peleus)
- 10mL volumet. pipette AS-class
- 20mL volumet. pipette AS-class
- 4 x 40mL beakers
- 4 x 50mL volumetric flask
- Test-tube rack & set of test-tubes
- Wooden Test-tube clamp
- Centrifuge w/ set of tubes
- Medicine dropper
- Bunsen burner
- Small spatula
- Protection glasses
- Paper towels
- Marker pen
- pH indictor paper
- Pasteur pipette

chemicals: Deionized water
- ≈20mL Hydrochloric Acid HCl (w = 32%)
- ≈15mL Nitric Acid HNO_3 (w = 65%)
- ≈15mL Ethanol C_2H_5OH (w = 96%)
- a pinch of Lead (II) Nitrate GR $Pb(NO_3)_2$ (w = 99%)
- a pinch of Potassium Chromate $CaCrO_4$ (w = 97%)
- a pinch of Dimethyl-Glyoxime $C_4H_8N_2O_2$ (w = 99%)
- sample of chemicals in test-tube



Relative mobility of selected ionic species at various pH and Redox-conditions (Förstner, 1989). Elements such as Cu, Pb, Ag, etc. form cations and are characterized by an increased motility at lower pH's. Elements such as V, U, Se, Si, As and Cr, are anions and reveal higher mobilities at pH-Werten >7. Ionic species such as Cr are subject to Redox-potential (Eh). Due to the oxidation of CrIII to CrII (CrO_4^{2-}), mobility increases with Redox-potential in basic media

Pb-specific reaction: $Pb^{2+}(aq) + K_2CrO_4(s)$ ↔ **black** $PbCrO_4(s) + 2K^+$
Ag-specific reaction: $Ag^+(aq) + 2NH_3$ ↔ $[Ag(NH_3)_2]^+(aq)$
Hg-specific reaction: $Hg^{2+}(aq) + 2NH_3(l)$ ↔ $[HgNH_2]^+(aq) + NH_4^+(aq)$
$[HgNH_2]^+(aq) + Cl^-(aq)$ ↔ **black** $HgNH_2Cl(s)$ also known as Calomel
Co-specific reaction: $Co^{2+}(aq) + 2NH_3(l) + 2H_2O(l)$ ↔ **green** $Co(OH)_2(aq) + 2NH_4^+(aq)$
Cu-specific reaction: $Cu^{2+}(aq) + 2NH_3(l) + 2H_2O(l)$ ↔ **blue** $Cu(OH)_2(aq) + 2NH_4^+(aq)$
Ni-specific reaction: $Ni^{2+}(aq) + 2NH_3(l) + 2H_2O(l)$ ↔ **white** $Ni(OH)_2(aq) + 2NH_4^+(aq)$
using Dimethyl-Glyoxime at pH 7 forms a **reddish**-like precipitate
$Ni^{2+}(aq) + 2C_4H_8N_2O_2(aq)$ ↔ $Ni(C_4H_8N_2O_2)_2(s)$

Procedure: Preparation of diluted acids/base and complexing agent (use 40mL beakers for the concentrated acids and base):
- to obtain a diluted HCl (4M) pipet the calculated volume of concentrated HCl into a 50mL volumetric flask, (use formula 14.1) and fill up with deionized water; shake well;
- repeat same procedure with concentrated HNO_3 to obtain a concentration of c = 2mol/L;
- repeat same procedure with concentrated H_2SO_4 to obtain a concentration of c = 2mol/L;
  Note: wear protection glasses & gloves at all times;
- dissolve the solid Dimethyl-Glyoxime sample in 96% ethanol using a 50mL volumetric flask;

Qualitative Analysis of sample solutions:
- fill sample of chemicals (obtained from tutors) with deionized water; already at this stage, the following hues may be indicators for:
  reddish ≈ indicator for Cobalt (Co);

bluish ≈ indicator for Copper (Cu);
greenish ≈ indicator for Nickel (Ni);

- dissolve any residue by gently heating test-tube with Bunsen burner; if residue is still present, add some drops of diluted HNO_3;
  Note: wear protection glasses & gloves at all times; work under aspirator (be aware that evaporation may occur in an explosive manner)!
- redistribute the dissolved content evenly into two different test tubes.

Checking for Ni^{2+}, Co^{2+}, Cu^{2+} in aqueous solution (pH ≥7) - 1st test tube:

- extract a tiny amount from the 1st test tube and pipet it into an empty 3rd tube (use Pasteur pipette);
- add a few drops of concentrated NH_3 to a 3rd test tube (pH >7), shake well and confirm with indicator paper; at this stage, in the presence of any of the above heavy metals, the following colorimetric reaction should occur:
  i) dark **green** is the evidence for Cobalt (**Co^{2+}**);
  i) deep **blue** is the evidence for Copper (**Cu^{2+}**);
  i) a white precipitate may form; in order to test for **Ni^{2+}-**ions, add few drops of Dimethyl-Glyoxime solution. A sudden change from colourless to a deep **red** is the finale proof.


aqueous solutions of dissolved ionic metals

Checking for Ag^{+}, Pb^{2+}, Hg^{2+} in aqueous solution – continue with 1st test tube:

- pipet a tiny amount from the 1st test tube into a centrifuge-tube, and add some drops of diluted 4M HCl (pH <7); stop adding HCl once no extra precipitate forms (the centrifugation will do the rest);
  Note: to counterweight the cartridge of the centrifuge, fill a 2nd centrifugation tube up to the same liquid level; this avoids asymmetrical stresses acting onto the rotor of the centrifuge;
- spin for approx. 2-3mins at 3k-4k/mins, and extract liquid phase into a standard 4th test-tube;

testing the liquid phase for **Pb^{2+}** ions:

- add a pinch of K_2CrO_4 to the liquid phase to test for lead; if no lead is present, crosscheck reaction by adding add some $Pb(NO_3)_2$ (sample should form a **black** precipitate when lead is introduced);

testing the solid phase for Ag^{+} and Hg^{2+}:

- wash precipitate with 0.5M HCl; add a few drops of water and boil – if necessary centrifuge while still hot; time allowing, repeat procedure to enrich precipitate;
  Note: be aware that centrifugation are not heat-resistant and might crack during heating;
- add a few drops of concentrated NH_3 to the precipitate (pH >7); in presence those two heavy metals, the following colorimetric reaction should occur:
  i) **black** precipitate in presence of Mercury (**Hg^{2+}**);
- i) if now reaction occurs add few drops of 4M HCl to redissolve the precipitate; a whitish, non-transparent reaction indicates the presence of silver ions;

.... at this point we terminated the analysis, since all major compounds involved have been detected ….

Results and Evaluation: Based on the observed reactions, the sample analysed contained traces of Co, Ni, & Hg

Formula 14.1: ($c_1 \cdot V_1 = c_2 \cdot V_2$)

$$V_{conc.} = \frac{c_{diluted} \cdot V_{diluted}}{c_{concentrated}}$$

c, concentration [mol/l]
V, volume [L]

Results of Dilutions (indicated in grey):

	Diluted		Concentrated	
	c [mol/L]	V [mL]	c [mol/L]	V [mL]
HCl	4	50	10.2*	≈20
HNO_3	2	50	14.35*	≈7
H_2SO_4	2	50	18.01*	≈5.6

(*) concentrations obtained from data sheet, see appendix

Experiment 15: Chromatography Day 9: 12th of March 1998

Purpose: Verification of the labeled amount [mg] of Acetylsalicylic Acid content (ASA) in a brand-type Aspirin pill. Reference samples of ASA with gradually increasing mass percentage are used to estimate the quantity of ASA contained in the brand-type pill.

material: Pipette filler (Peleus)
10mL measuring pipet AS-class
50mL volumet. pipette AS-class
5 x 2μL Volumetric capillary
100mL volumetric flask
5 x 25mL volumetric flask
Chromatographic chamber (TLC)
TLC plate (20 x 20 cm) coated w/ silicagel $60F_{254}$
small mortar w/ pestle
Filter paper
Pencil
UV-Analyzer

chemicals:
≈3g AcetylSalicylic Acid $C_6H_9O_4$ (ASA)
≈40mL Cyclohexane C_6H_{12} (w = 99%)
≈50mL Chloroform $CHCl_3$ (w = 99%)
≈10mL glacial Acetic Acid CH_3CO_2H (w = 100%)
≈125mL Acetone
Aspirin tablet of Bayer (labelled content: 320mg/tablet)

Procedure: Preparation of Thin Layer Chamber:

- line TLC chamber with filter paper and

Preparation of cyclohexane carrier medium:

- pipet 50mL of chloroform into the 100mL volumetric flask;
- add 10mL of Acetic Acid (w = 100%), and fill up the rest (40mL) with Cyclohexane; shake well;
- pour solution into TLC chamber and close lid;

Preparation of brand-type sample ASA pill:

- grind ASA pill, place into a 25mL volumetric flask and fill flask with pure acetone (shake well);

Preparation of reference ASA solution:

- weigh 0.25, 0.5, 0.75, 1g of the ASA powder into 4 separate 25mL volumetric flasks;
- fill up each flask with Acetone and shake well;

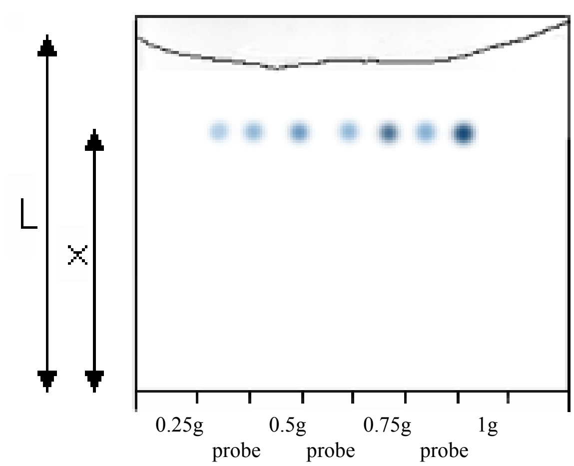
Applying samples onto TLC plate:

- divide TLC plate into sections as shown below;
- dip capillary pipette into reference solution and place at appropriate spot
  Note: use one pipet for each reference solution only; apply pipet perpendicularly, make sure that liquid contained in pipet is completely absorbed by the TLC plate;
- dip capillary pipette into brand-type ASA test solution and likewise place it at the indicated spots;
- slide TLC plate into chamber and close firmly;
- once the carrier medium reached the upper limit (approx. after 2 hours) mark migrating border with pencil and allow solvent to vent off under aspirator (carrier medium evaporates quickly);
- place dried plate under UV light, measure the distances from the starting point of both carrier medium (upper limit) and ASA samples (reference and brand-type samples) and calculate the retention factor (R_F);

Results and Evaluation: ASA content printed on the box met the requirements. According to hue of spot, the mass Percentage of probe must be in-between 0.25 and 0.5g (tending more towards the lower end).

	x : l	R_F
RS $_{0.25g}$	12.1 : 15.4	0.79
TS	12.1 : 15.3	0.79
RS $_{0.5g}$	11.8 : 15.1	0.78
TS	12.0 : 15.0	0.8
RS $_{0.75g}$	11.7 : 14.9	0.79
TS	11.8 : 14.9	0.79
RS $_{1g}$	11.6 : 15.0	0.79
		0.787

RS....Reference Solution
TS....Test Solution



As for reference, R_F value should be identical with other groups

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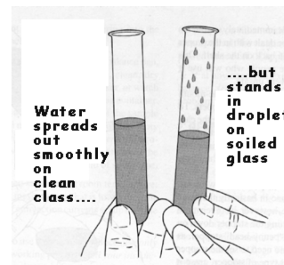
Förstner, U.; Kersten, M.; Wienberg, R.; 1989; Geochemical processes in landfills. In: Baccini, P. (Hrsg.): The Landfill - Reactor and Final Storage. Lecture Notes in Earth Sciences 20, S. 39-81. Berlin, Heidelberg,
http://home.t-online.de/home/r.wienberg_umwelttechnik/alt3_21.pdf

Laboratory Utensils (and Techniques):

Handling of Chemicals: No matter what chemicals are used (rare or common), what amount is needed, or what the form of the material (pure or in solution, liquid, solid) some general rules should be followed:

- No material, no matter how much is supplied, should be wasted.
- Always be sure that the chemical withdrawn from the bottle is **exactly** the one you need.
- Read the label carefully **before** you take any sample.
- Nothing should be done to change the purity of the material in the stock bottle - once it is out of the bottle, it is out - any excess should **not** be put back into the bottle.
- Any excess material should be disposed of in a safe, responsible manner.
- Do not stuck your nose into bottles to catch the contents smell.
- Wear protection gloves and glasses at any time.

Cleaning of Lab-Utensils Before using any volumetric utensils (buret, pipet, graduated cylinders, test-tubes etc.), they must be thoroughly cleaned (so that no water droplets adhere to the inner walls), then rinsed with the solution that is to be measured (in burets: with closed stopcock), so that the entire inner surface comes into contact with the liquid.

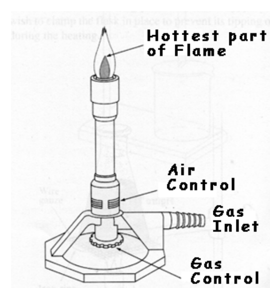


The rinsed liquid is then discarded into the sink or other appropriate waste container.

Buchner Funnel: A glass or porcelain funnel with a ceramic filtering plate instead of a paper filter; used for suction filtration - see there for further details.

Burets: When repeated measurements of nonround volumes such as 19.57mL are needed, a buret is the most common choice. Burets are long graduated glass cylinders, available in many sizes from 1 to 100mL. At the bottom, a buret has a glass or plastic stopcock for controlling the flow rate of the liquid.

Bunsen Burner: Typical lab-burners use natural gas (mostly methane CH_4) as their fuel. The burner is connected to the gas source by means of a flexible hose. Open gas valve fully, then wait a few seconds for the gas to fill the line. A match or striker is used to light the flame at the burner head.



Adjusting the heat of the flame: The air is controlled by opening or closing a series of holes at the base of the burner tube. The gas control should be open half way.

- A “lean“ flame (too much air) will give a roaring noise and will easily blow out.
- A “rich“ flame (too little air) will be yellow.
- The hottest point of the flame is just above the tip of the inner dark blue cone.

Note: Care must always be taken when heating a liquid in a test tube. The long, narrow shape is like that of a canon, and hot material can be “shot“ from the test tube for quite a distance of care is not taken. For this reason, be sure that the tube is heated slowly, that the center of the tube is heated (near the surface of the material, not the very bottom), and that the mouth of the tube is not pointed at anyone during the heating process.

Cation Exchanger:

Centrifuge:

Chromatograph:

Distillation Apparatus: If solutions consisting of two or more compounds need to be separated; one of the components of the solution will be more volatile (evaporating more rapidly then the other). The solution is heated until it begins to boil. A stream of cold water running through the condenser causes the vaporized material to be condensed back into a liquid so that it can be trapped in a receiver-flask.

- Open outlet at the other end of the condenser is necessary (filled with absorber chemicals or other filters) to allow increasing pressure to escape

Erlenmeyer Flask: A flask with a narrow glass neck which gradually opens to the bulge bottom part. Can be made to fit with a glass stopper or simply open.

Flat-Pan Balance: These types of top-load balances have a single where the object is weighed. This type of balance generally reads to the nearest 0.01g depending on the model. Usually the mass is obtained directly from a digital readout display.

To operate a balance:

- A balance must kept clean.
- Never weigh any chemicals directly on the balance pan; use a beaker, watch glass and add the sample to weigh in there. The mass of the sample is then equal to the total mass minus the mass of the empty container (Weighing by Difference).
- Objects to be weighed should be at room temperature; a hot, cold object will give a mass reading that is lower/higher than the correct value (convection/condensation effects).
- Allow to warm up before use; leave balance on if you are sure you need it more often; heat-up times can be time-consuming and may delay your work.

Graduated Cylinder: For volume measurements to the nearest 1mL or 0.1mL. These are tall glass or plastic cylinders with a wide base; commonly found in sizes of 10, 25, 50, 100, 250mL, even up to 5L.

They are usually calibrated to contain a certain volume of liquid, and are often marked TC to indicate this. Glassware marked in this way will contain the specified amount, but will deliver less than this when the contents are poured out (small amounts of liquid remain in the container - adhesion); if you must deliver a precise amount of liquid then a pipet or buret must be used.

Indicator: A weak organic acid or base that change color when it goes from its acid to its base form; i.e. acid-base neutralization (an acid-base indicator) or from its oxidized to its reduced form (a redox indicator);

Phenolphthalein changes sharply its color once the pH is slightly above 8. Once the stoichiometric point is passed, there is a sudden rise through pH=7 as the OH^- molarity increases sharply; i.e. once the anylate solution starts to become increasingly basic.

Other indicators (not used here in these experiments) are Thymol blue (pH 1.2-2.8), Bromophenol blue (pH 3.0-4.6), Methyl orange (pH 3.1-4.4), Methyl red (pH 4.2-6.3), Chlorophenol blue (pH4.8-6.4) Bromothymol blue (pH 6.0-7.6), Cresol red (pH 7.2-8.8).

Lab-Jack: Height adjustable, small table hoist (lifting jack).

Magnetic Stirrer: Device to heat up and simultaneously stir liquids loaded onto the hot-plate. Comes along with magnetic rod which placed into the container to be stirred (is driven by a rotating magnet beneath the hot-plate).

Pipette Filler (Peleus Rubber Bulb): On-handed palm bulb to suck up liquids into a pipet. Three valves allow easy handling of extraction and dosage of liquids.

- Do not suck up small amounts of liquids into a pipet, sucking up air inevitably will force liquid within pipet into the rubber bulb. This can cause premature deterioration of the rubber, especially when handling strong acids or bases.
- Never lay down a loaded pipet with the Peleus ball attached (liquid flows back into the ball).

pH Meter: A device to measure of how acid or basic a solution is, with pH values below 7 being acidic, those above 7 basic (alkaline), and pH of exactly 7 being neutral.

Indeed pH meters do not measure exactly the pH ($-\log[H^+]$) but rather the activity of these ions, whether shielded by other ions or allowed to diffuse freely. General hints how to use a pH meter:

- Remove electrode from storage solution and rinse well with deionized water. Be careful not to touch, rub, or damage the thin, delicate glass membrane in the tip of the electrode.
- Place electrode in a buffer solution of high (basic) pH and adjust to stated pH.
- Remove the electrode from this solution, rinse the tip well with deionized water, and place it in a buffer solution of low (acidic) pH. Adjust meter to the appropriate value.
- If not automatic (electronic) repeat previous two steps until reading is stable.
- Remove electrode from the solution, rinse the tip well with deionized water, and place it in the solution whose pH is to be measured.
- Rinse electrode well after measurements with deionized water, and place it back in its storage container. *Be sure that the tip of the electrode does not dry out*.

Pipet: For volume measurements more precise than ±0.1mL. Pipets are useful for delivering "round" volumes such as 2, 5, 10, 25, 50, 100mL). It is a narrow glass or plastic tube tapering to a fine point at one end and having at least one calibration marking on it.

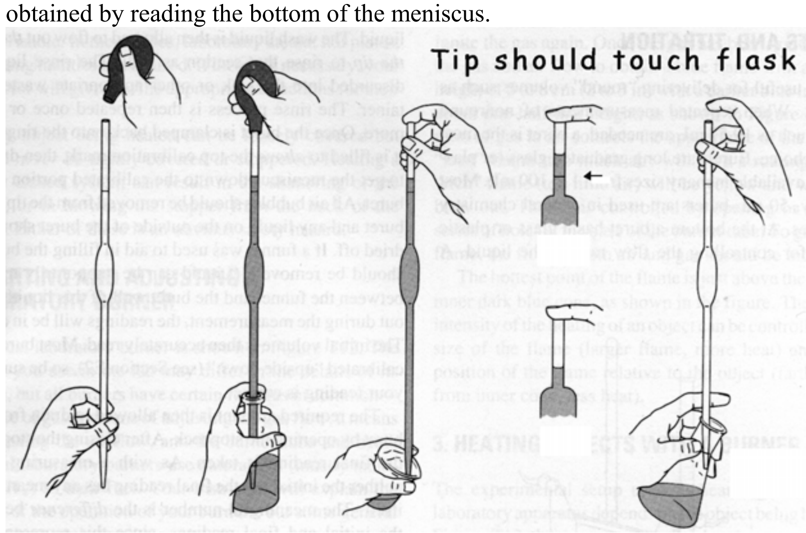
Volumetric P.: Often has a bulge in the center and has only one calibration marking on the upper part of the tube.

- Volumetric P. is calibrated to deliver exactly one specified amount (marked as TD or AS); it should be allowed to drain freely (wait 15secs after complete run-of) until no more liquid comes out.
- Any residual amount of liquid should not be blown or rinsed out.
- Cleaning pipets: see buret.

Mohr (Measuring) P.: A tube of constant diameter with markings along of its length; this implies that the more liquid is sucked in the "precise" the measured liquid is within the tube.

IMPORTANT: It is absolutely necessary to use a pipet bulb (Peleus bulb) to suck the liquid in the pipet; it is an extremely unsafe practice to use mouth suction for this.

Reading Meniscus: Volumetric glassware is always calibrated so that the correct reading will be obtained by reading the bottom of the meniscus.



Reflux Apparatus: If a reaction should be maintained while when one compound is more volatile than the other (evaporating more rapidly then the other). The solution is heated until it begins to boil. A stream of cold water running through the condenser causes the vaporized material to be condensed back into a liquid to fall back into the reaction flask.

- Open outlet at the other end of the condenser is necessary (filled with absorber chemicals or other filters) to allow increasing pressure to escape.

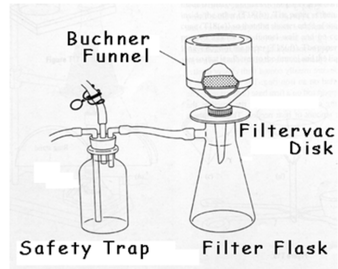
Separator Flask: Drop-shaped glass container with glass stopper on one and stopcock valve on the other; allows easy separation of liquids which are not mixable (hydrophobic and hydrophilic phases). It is mounted onto the stand with a ring-clamp to allow easy handling of stopcock.

Spectrometer: This instrument measures the intensity of light that passes through a solution contained in a sample container (cuvet). The intensity of the light can be related to the concentration of the species in the solution that absorbs the light. The fraction of light absorbed by a sample depends on the sample itself, the wavelength, the concentration of the absorbing species, and the length of the light path through the sample (Beer-Lambert law).

- The spectrometer must be warmed up (14 to 20mins) to give stable readings.
- Cuvets must be clean. When cleaning, be sure that no scratches are made on the scanning window (since scratches will scatter light).
- The instrument must be properly set to zero percent transmittance (infinite absorbence) and to 100 percent transmittance (zero absorbence) before any reading are made on your unknown sample; scaling can also be achieved by using a reference sample to which to adjust to.

Stand & Clamps: Tripod with clamps which can be fastened onto the holdfast; to hold burets, thermometers and other devices.

Suction Filtration: If gravity filtration with filter paper is not a suitable method (speed of filtration slows down); to speed the filtration, a vacuum or suction filtration is often done. A Buchner filter, a filtervac sealing ring, a filter flask and a water-jet driven pump are needed to assemble it. The filter flask looks like a typical Erlenmeyer flask, except that it is made of very thick glass and has a glass-sidearm near the top. The entire apparatus is connected to a source of vacuum using heavy-walled rubber or plastic tubing. The vacuum source may be an aspirator that uses water to generate the vacuum (water-jet pump) or it may be a vacuum pump.



Often a safety trap, placed between the source and the filter flask, is used for the actual filtration (helps to prevent the backup of unwanted material into the system).

Test-Tube Rack: Stand to hold empty and filled test-tubes vertically.

Thermometer: A set of mercury thermometers are available covering a range of 0 to 30°C or starting from 70 to 150°C.

Watch glass: Cone-shaped glass bowl.

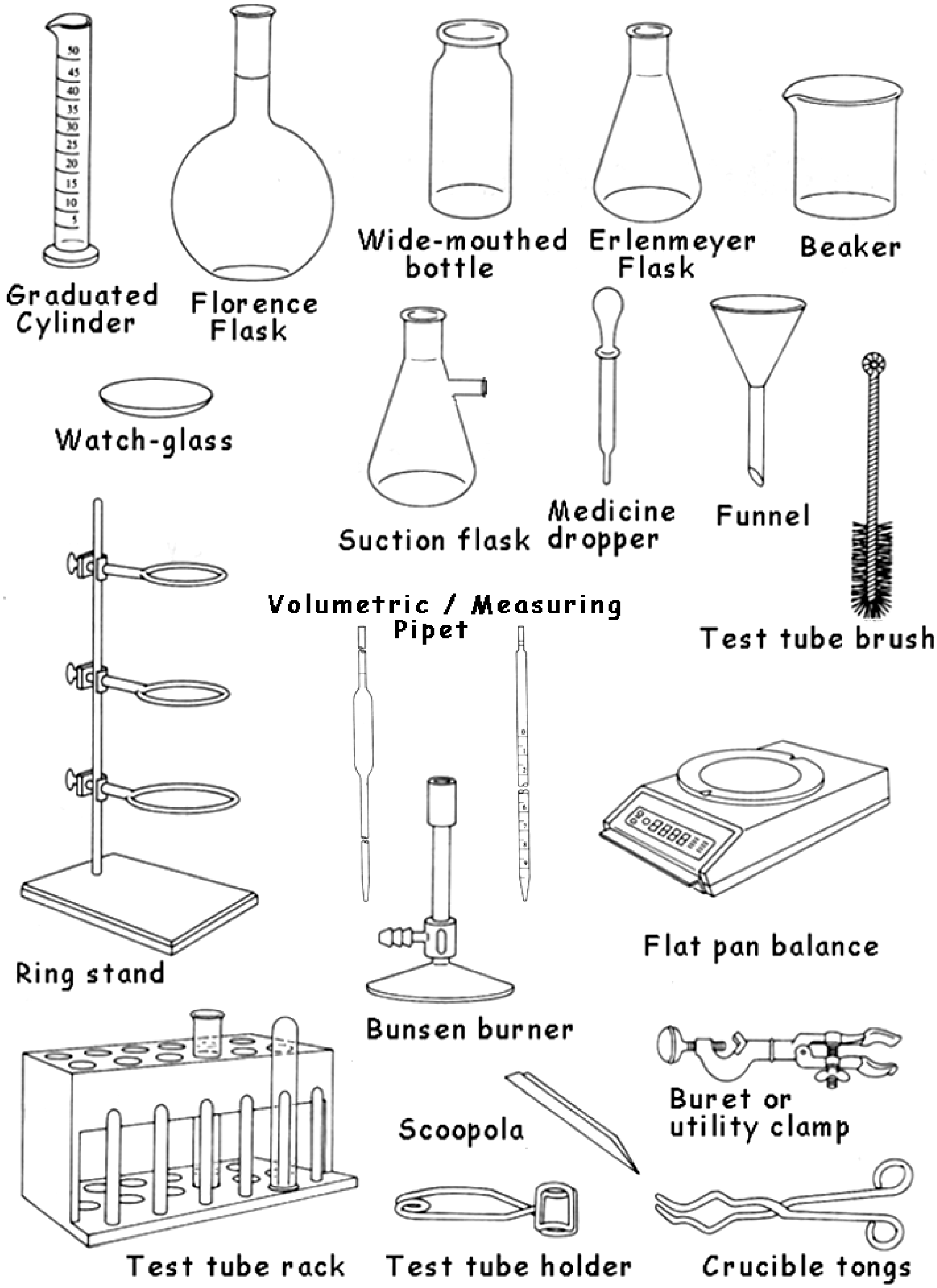
Vaccum Dessicator: A large glass container used to place Bunsen burner treated crucibles, etc. with a removable top part. The larger bottom part is divided into two chambers separated with a perforated ceramic tray.

The lower part s filled with hygroscopic substances (silicagel with $CoCl_2$ as indicator - blue when active, red when saturated with water). Objects to be cooled off are placed on top of the tray. The lid is equipped with a stopcock to allow ventilation and the use of a vacuum pump.

Volumetric Flask (**Florence** F.): For volume measurements more precise than ±0.1mL. Volumetric flasks are useful for delivering “round“ volumes such as 25, 50, 100, 250, 500mL. Has a narrow glass or plastic neck with usually one calibration marking on it and a bulge bottom.

Reading Meniscus: Volumetric glassware is always calibrated so that the correct reading will be obtained by reading the bottom of the meniscus.

Overview of utensils:



Formula 1.1, 4.1, 6,1, 7.1:

$c_{Sln} =$	n_{Ste}/V_{Sln}	extended with: $n_{Sln} = m_{Ste}/M_{Slt}$ n, molar amount V, volume	 [mole] [L]
$c_{Sln} =$	$m_{Ste}/(V_{Sln} \cdot M_{Ste})$	extended with: $m_{Slt} = w_{Slt} \cdot m_{Sln}/100$	
$c_{Sln} =$	$\frac{w_{Ste} \cdot m_{Sln}}{100 \cdot V_{Sln} \cdot M_{Ste}}$	extended with: $m_{Sln} = \rho_{Sln} \cdot V_{Sln}$	
$c_{Sln} =$	$\frac{w_{Ste} \cdot \rho_{Sln}}{100 \cdot M_{Ste}}$	w, mass percentage ρ, density M, molar mass	[%] [g/L] [g/mol]

Sln = solution;
Ste = solute

Formula 4.3:

1mL Titriplex solution (V_{EDTA}, c = 0.1mol/L) fixes the equivalent of 5.585mg $FeCl_3 \cdot 6H2O$			
m =	$\frac{V_{EDTA} \cdot 5.585E^{-3}}{V_{diluted}}$	$V_{diluted}$, diluted Titriplex solution	
m =	$\frac{V_{EDTA} \cdot 5.585E^{-3} \cdot 10}{V_{diluted}}$	10, dilution factor of Titriplex solution	
m =	$\frac{V_{EDTA} \cdot 55.85E^{-3}}{V_{diluted}}$	converted to Fe-ions with $M_{FeCl3 \cdot 6H2O}/M_{Fe}$	
$m_{Fe} =$	$\frac{V_{EDTA} \cdot 55.85E^{-3} \cdot M_{FeCl3 \cdot 6H2O}}{V_{diluted} \cdot M_{Fe}}$	V_{EDTA}, volume M, molar mass $V_{diluted}$, volume	[L] [g/mol] [L]

Formula 5.1: $n_{Zn} + ½n_{O2} = n_{ZnO}$

$m_{Zn} =$	$n_{Zn} \cdot M_{Zn}$	$n_{Zn} = n_{ZnO}$	
$m_{Zn} =$	$n_{ZnO} \cdot M_{Zn}$	extended with: $n_{ZnO} = m_{ZnO}/M_{ZnO}$	
$m_{Zn} =$	$\frac{m_{ZnO} \cdot M_{Zn}}{M_{ZnO}}$	m_{ZnO} w/o dish = $m_{dish+ZnO}$-$m_{empty\ dish}$	
$m_{Zn} =$	$\frac{(m_{dish+ZnO} - m_{empty\ dish}) \cdot M_{Zn}}{M_{ZnO}}$	n, molar amount M, molar mass m, mass	[mole] [g/mol] [g]

Formula 7.3:

$m_{NaAc} =$	$n_{NaAc} \cdot M_{NaAc}$	extended with: $n = c \cdot V$ n, molar amount	 [mol]
$m_{NaAc} =$	$c_{NaAc} \cdot V_{NaAc} \cdot M_{NaAc}$	c, concentration V, volume M, molar mass	[mol/L] [L] [g/mol]

Formula 10.5:

$K_C =$	$\frac{c_{IBE} \cdot c_{H2O}}{c_{HAc} \cdot c_{IB}}$	extended with: $n = c \cdot V$	
$K_C =$	$\frac{(n_{IBE}/V) \cdot (n_{H2O}/V)}{(n_{HAc}/V) \cdot (n_{IB}/V)}$	constant V is canceled	
$K_C =$	$\frac{n_{IBE} \cdot n_{H2O}}{n_{HAc} \cdot n_{IB}}$	n, mole amount	[mol]

Formula 8.3: (equation is based on reaction product, water)

$C =$	$m_{solution} \cdot s$	s, specific heat capacity; $s_{H2O} = 4.184$	[J/(g·°C)]
$Q_{Solution} =$	$C \cdot \Delta T$	m, mass	[g]
$Q_{Solution} =$	$m_{solution} \cdot 4.184 \cdot \Delta T$	extended per mole of product n, molar amount	[mol]
$Q_{Solution} =$	$\frac{\Delta T \cdot m_{solution} \cdot 4.184}{n_{solution}}$	T, temperature Q, energy	[°K] [kJ]

Formula 11.1: ($n_{NaNO3} \rightarrow n_{Na+} + n_{NO3}^{-}$)

$n_{NaNO3} =$	m_{NaNO3}/M_{NaNO3}	converted to n_{NO3}^{-}	
$n_{NO3}^{-} =$	$\frac{m_{NaNO3} \cdot M_{NO3-}}{M_{NaNO3}}$	converted to β_{NO3}^{-} n, molar amount	[mol]
$\beta_{NO3-} =$	$\frac{m_{NaNO3} \cdot M_{NO3-}}{M_{NaNO3} \cdot V_{100}}$	including dilution; M, molar mass	[g/mol]
$\beta_{NO3-} =$	$\frac{m_{NaNO3} \cdot M_{NO3-} \cdot V_{pipet}}{M_{NaNO3} \cdot V_{100} \cdot V_{500}}$	β, mass concentrn. V, volume	[g/L] [L]

Formula 12.1:

10mL EDTA solution (V_{EDTA}, c = 0.01mol/L) fixes the equivalent of $0.1E^{-3}$ mol CaO			
$c_{H2O} =$	$\frac{c_{Titrant} \cdot V_{Titrant}}{V_{Water\ Sample}}$	$c_1 \cdot V_1 = c_2 \cdot V_2$ converted into g/mol	
$\beta_{CaO} =$	$\frac{c_{Titrant} \cdot V_{Titrant} \cdot 56.08}{V_{Water\ Sample}}$	$M_{CaO} = 55.08$ g/mol multiplied with 1L β, mass conc.	[g/mol]
$m_{CaO} =$	$\frac{c_{Titrant} \cdot V_{Titrant} \cdot 56.08}{V_{Water\ Sample}}$	c, concentration V, volume m, mass	[mol/L] [L] [g]

Formula 12.2:

1mg of CaO in 100mL is the equivalent of 1° dH			
$dH_{sample} =$	$\frac{m_{CaO} \cdot 100E^{-3}}{1E^{-3}}$		
$dH_{sample} =$	$m_{CaO} \cdot 100$	m, mass dH, hardness	[g] [°]

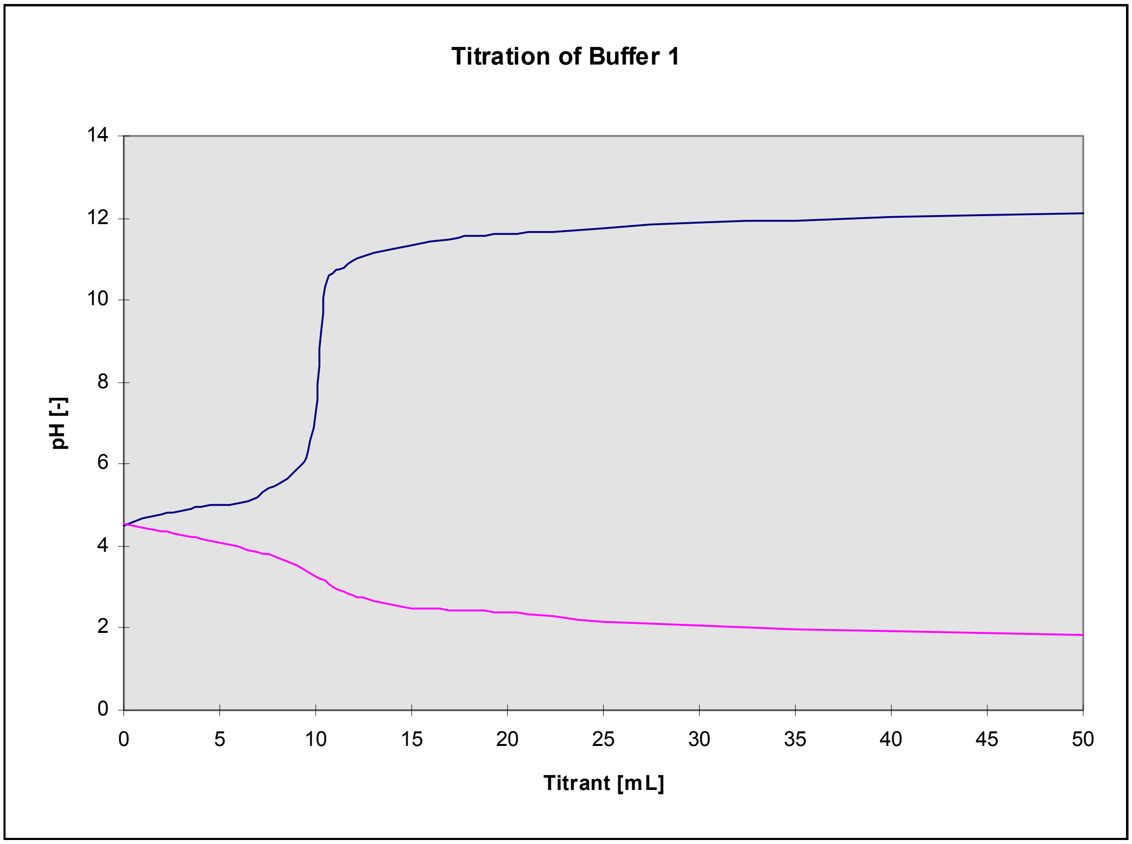
Formula 13.2:

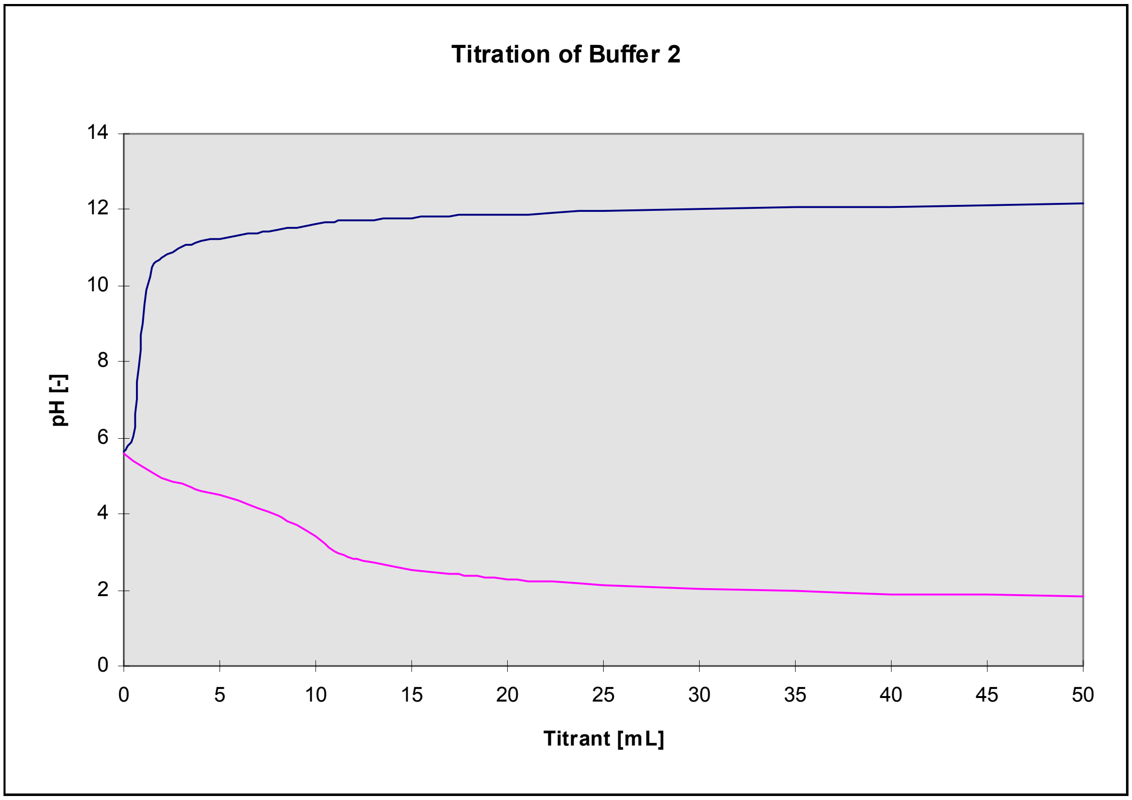
$n_{EDTA} =$	$c_{EDTA} \cdot V_{EDTA}$	EDTA = titrant	
$n_{Ba-left} =$	$n_{Ba} - n_{EDTA}$	$0.1E^{-3}$, initial n_{Ba}	[mol]
$n_{Ba-left} =$	$(c_{BaCl2} \cdot V_{BaCl2}) - (c_{EDTA} \cdot V_{EDTA})$	$n_{Ba-left}$ in Analyte	
$n_{Ba-left} =$	$\frac{[(c_{Ba} \cdot V_{Ba}) - (c_{Titrant} \cdot V_{Titrant})] \cdot V_{analyte}}{V_{dil.Analyte}}$	$n_{Ba-left}$ of undiluted Analyte	
$n_{Ba-left} =$	$\frac{[(c_{Ba} \cdot V_{Ba}) - (c_{Titrant} \cdot V_{Titrant})] \cdot V_{Analyte} \cdot V_{Probe}}{V_{dil.\ Analyte} \cdot V_{ion-Exchange}}$	$n_{Ba-left}$ of entire Probe	
$c_{Ba-left} =$	$\frac{[(c_{Ba} \cdot V_{Ba}) - (c_{Titrant} \cdot V_{Titrant})] \cdot V_{Analyte} \cdot V_{Probe}}{V_{dil.\ Analyte} \cdot V_{ion-Exchange} \cdot V_{Probe}}$	divided by V_{Probe} to obtain mol/L	
$Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s)$ according to the equation above, Ba^{2+} reacts with sulfate to form a solid precipitate; remaining Ba^{2+} reacts with EDTA; once no Ba^{2+} is left, additional EDTA changes color of buffer; i.e. indirect indicator of SO_4^{2+}			
$c_{SO4--} =$	$\frac{[(c_{Ba} \cdot V_{Ba}) - (c_{Titrant} \cdot V_{Titrant})] \cdot V_{analyte}}{V_{dil.\ Analyte} \cdot V_{ion-Exchange}}$	c, concentration V, volume m, mass	[mol/L] [L] [g]

Appendix A – Experimental Data Sheet:

Experiment 7 - Titration of two buffers (B-1 & B-2) with self-made reference-base and -acid:

	pH of Buffer-1 (1_{HAc} : 1_{NaAc})		pH of Buffer-2 (1_{HAc} : 10_{NaAc})	
Titrant $V_{Titrant}$ [ml]	NaOH (c = 0.0980mol/l)	HCl (c = 0.0988mol/l)	NaOH (c = 0.0980mol/l)	HCl (c = 0.0988mol/l)
0	4.52 (22.1°C)	4.54 (22.0°C)	5.64 (22.3°C)	5.60 (22.7°C)
0.5			6.04	5.38
1	4.70	4.45	9.01	5.22 (22.7°C)
1.5			10.51	5.08 (22.6°C)
2	4.78	4.36	10.75	4.97
2.5				4.86
3	4.85	4.28	11.04	4.78
3.5				4.71
4	4.94	4.18	11.20	4.62
5		4.10		4.48
6	5.05	4.00	11.31	4.33
7	5.17 (22.1°C)	3.86		4.17
7.5	5.41 (22.2°C)	3.80		
8	5.52	3.72		3.97
8.5	5.66	3.64		3.87
9	5.89	3.53		3.73
9.5	6.15 (22.2°C)	3.39		3.56
10	7.26 (22.3°C)	3.28 (22.0°C)	11.62	3.39
10.5	10.33			3.21
11	10.71	3.00 (22.1°C)		3.04
11.5				2.92
12	10.99	2.81		2.83
13	11.17	2.68		2.70
14		2.59		2.60
15	11.36	2.50	11.78	2.53
16				2.46
17				2.41
18				2.37
20	11.63	2.38	11.88	2.28
25	11.77	2.15		2.15
30	11.88	2.05 (22.1°C)	12.01	2.05
35	11.95	1.98 (22.2°C)		1.96
40	12.01	1.92	12.09 (22.3°C)	1.90
45	12.06	1.88 (22.2°C)		1.87
50	12.10 (22.3°C)	1.82 (22.3°C)	12.17 (22.4°C)	1.83 (22.6°C)


Titration of Buffer 1
pH [-]
Titrant [mL]
0
2
4
6
8
10
12
14
0
5
10
15
20
25
30
35
40
45
50


Titration of Buffer 2
pH [-]
Titrant [mL]
0
2
4
6
8
10
12
14
0
5
10
15
20
25
30
35
40
45
50

Experiment 8 - Enthalpy of Reaction:

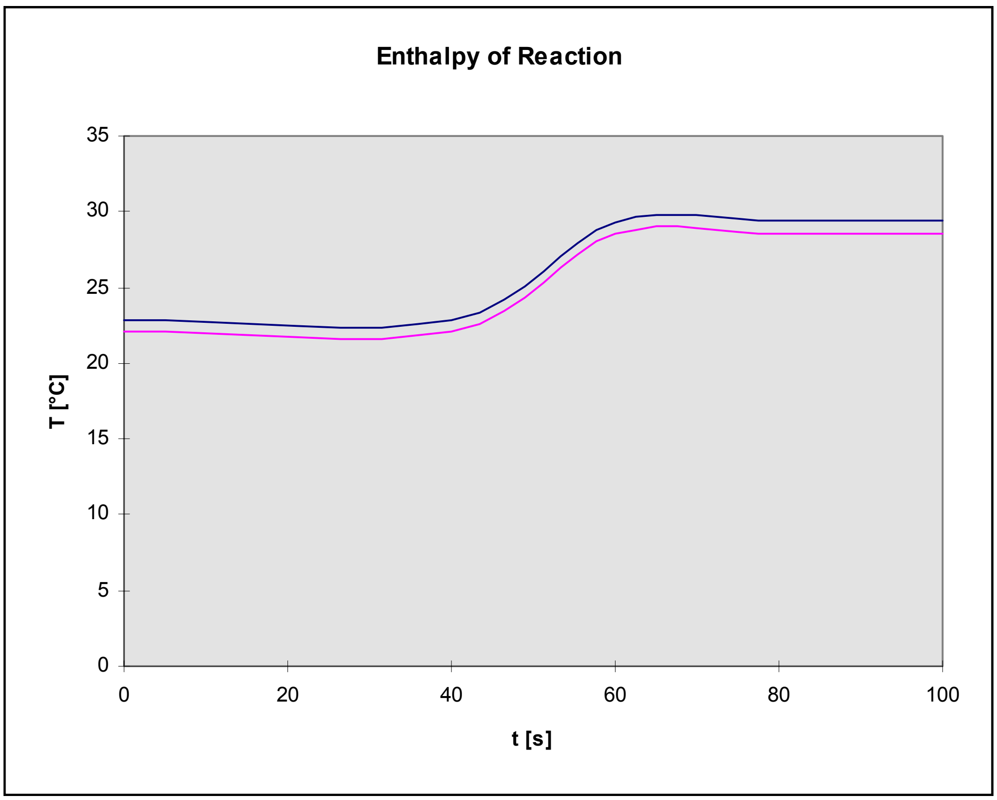
Approximation of density and concentration values of HCl and NaOH
Results indicated in gray:

averaged values of lines:	HCl (c = 0.988mol/l)		NaOH (c = 0.980mol/l)	
	ρ [g/l]	c [mol/l]	ρ [g/l]	c [mol/l]
1	1015*	0.9391*	1040*	0.9710*
2	1020*	1.227*	1045*	1.097*
3: (1+2)/2	1017.5	1.0831	1042.5	1.034
4: (1+3)/2	1016.3	1.01111	1041.3	1.0025
5: (1+4)/2	1015.6	0.975	1041.6	0.98675
6: (1+5)/2			1040.3	0.9788
7: (4+5)/2	1015.9	0.9309		
8: (4+7)/2	1016.1	1.0021		
9: (7+8)/2	1015.9	0.9886		

*) values obtained from data sheets

Temperatures recorded:

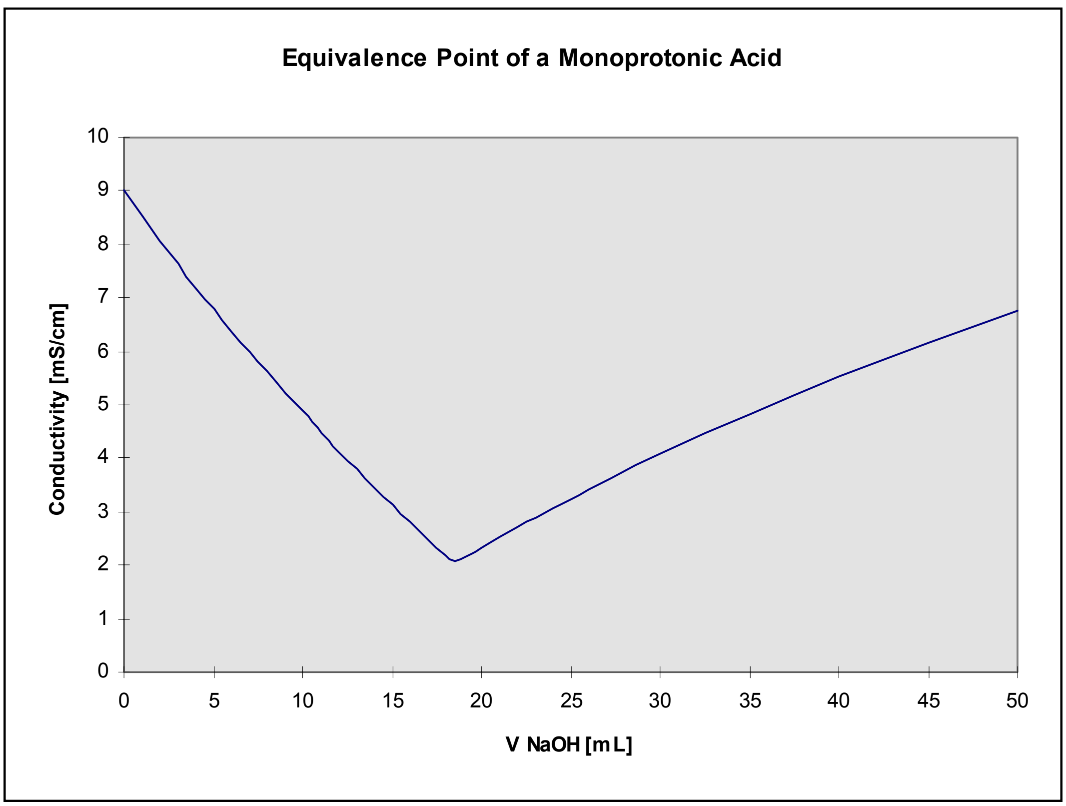
Trial	1 - T [°C]	2 - T [°C]
t_0	22.80	22.15
t_{20sec}	29.30	28.50
t_{40sec}	29.40	28.60
t_{1min}	29.40	28.50
t_{4mins}	29.00	28.00
t_{7mins}	28.50	27.80



Experiment 9 - Determining the Equivalence Point of a Monoprotic Acid:

Results of Titration (conductivity depression indicated in gray):

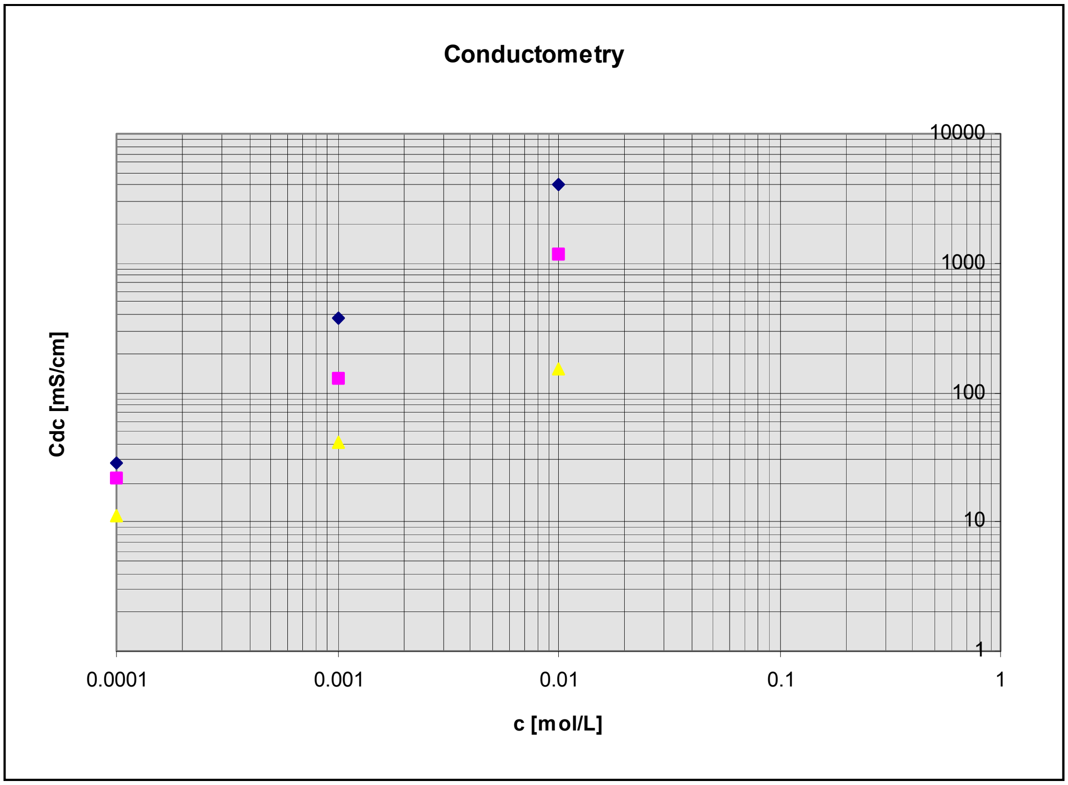
NaOH $V_{Titrant}$ [ml]	Conductivity [mS/cm]	NaOH $V_{Titrant}$ [ml]	Conductivity [mS/cm]
0	9.01 (22.7°C)	15	3.12 (21.9°C)
1	8.52	15.5	2.95
2	8.09 (22.7°C)	16	2.81
3	7.65 (21.8°C)	16.5	2.64
4	7.185	17	2.48
5	6.78	17.5	2.33
6	6.375	18	2.19
7	6.00	18.5	2.07
8	5.62	19	2.14
9	5.21	19.5	2.23
10	4.88	20	2.34
10.5	4.67	21	2.52
11	4.46	22	2.70
11.5	4.33	23	2.90
12	4.13 (21.8°C)	24	3.07
12.5	3.94 (21.9°C)	25	3.23
13	3.80	30	4.07
13.5	3.63	35	4.82
14	3.45	40	5.52
14.5	3.28	45	6.16
15	3.12 (21.9°C)	50	6.75 (21.9°C)



Experiment 9 -Conductance of Aqueous Solutions:

Measurements of conductance

concentration		0.01 [mol/L]	0.001 [mol/L]	0.0001 [mol/L]
[µS/cm]	HCl	4010	374	22.9
	NaCl	1156	129.0	21.6
	HAc	153.2	40.8	11.0



Experiment 10 - Determining Equilibrium Constant:

Approximation of density and concentration values of HCl
Results indicated in gray:

averaged values of lines:	HCl (c = 0.988mol/l) ρ [g/l]	c [mol/l]
1	1075*	15.485*
2	1080*	16.47*
3: (1+2)/2	1077.5	15.978
4: (2+3)/2	1078.8	16.224
5: (3+4)/2	1078.1	16.101
6: (3+5)/2	1077.8	16.039

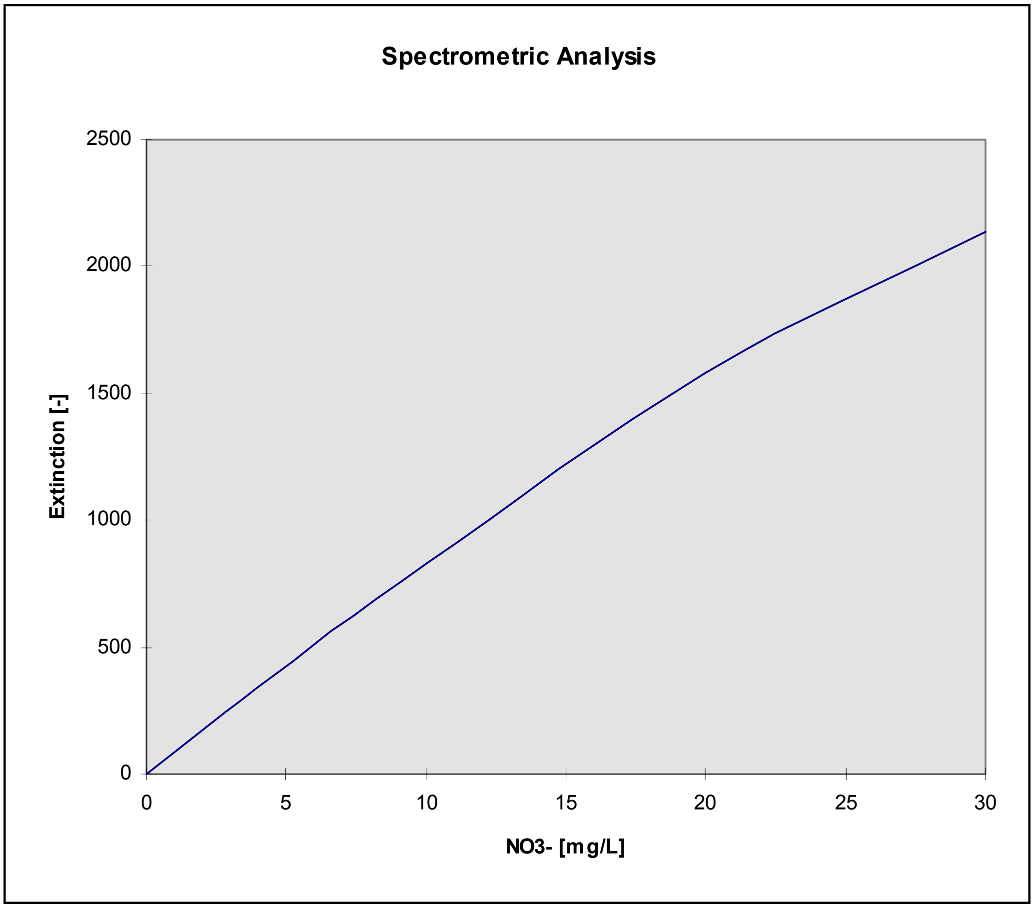
*) values obtained from data sheets
Approximation of density and concentration values of HCl
Results indicated in gray:

Experiment 11 - Spectrophotometric Analysis:

Readings obtain from the spectrophotometer of calibration and test samples:

Calibration probes	$NaNO_3$ content [mg/L]	NO_3^- [mg/L]	Extinction [-]
1st probe	15	30	2137
2nd probe	10	20	1630
3rd probe	5	10	783
4th probe	2	4	342
5th probe	0	0	0
Test probes			
tab-water		522	522
Tutor's probe		10	817
Dr. Malissa's probe		13.75 x 4*	1039

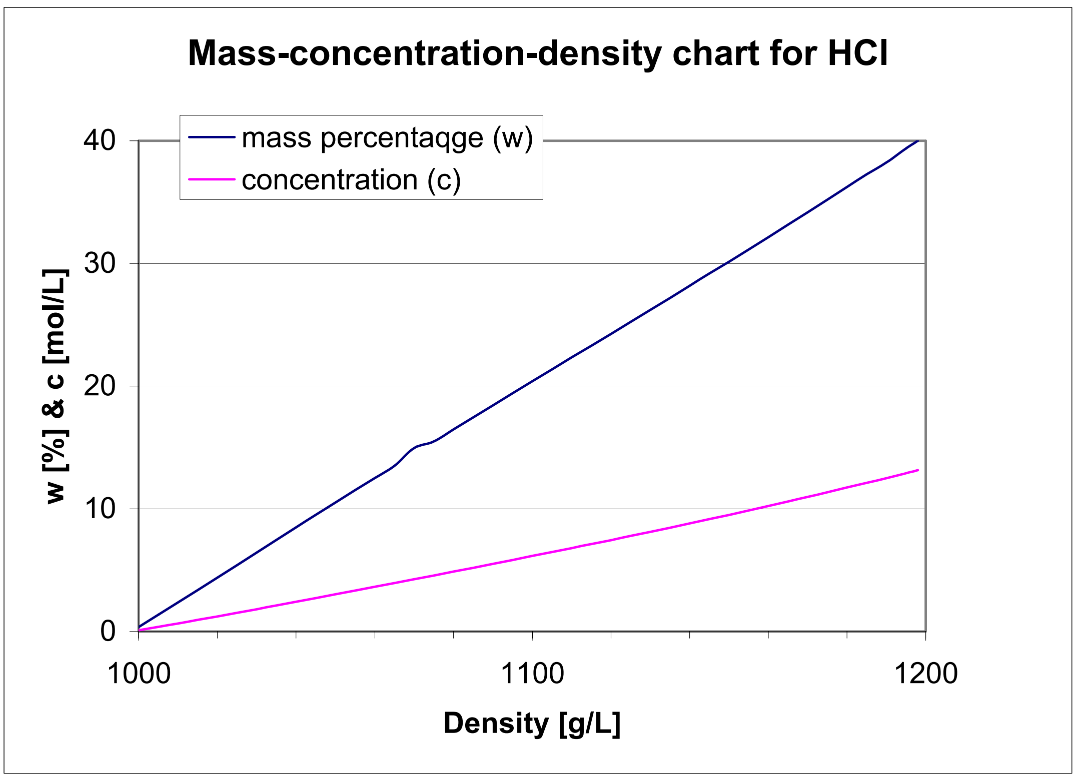
(*) sample has been given already as a 1:4 diluted sample



Appendix B – Mass-concentration-density charts:

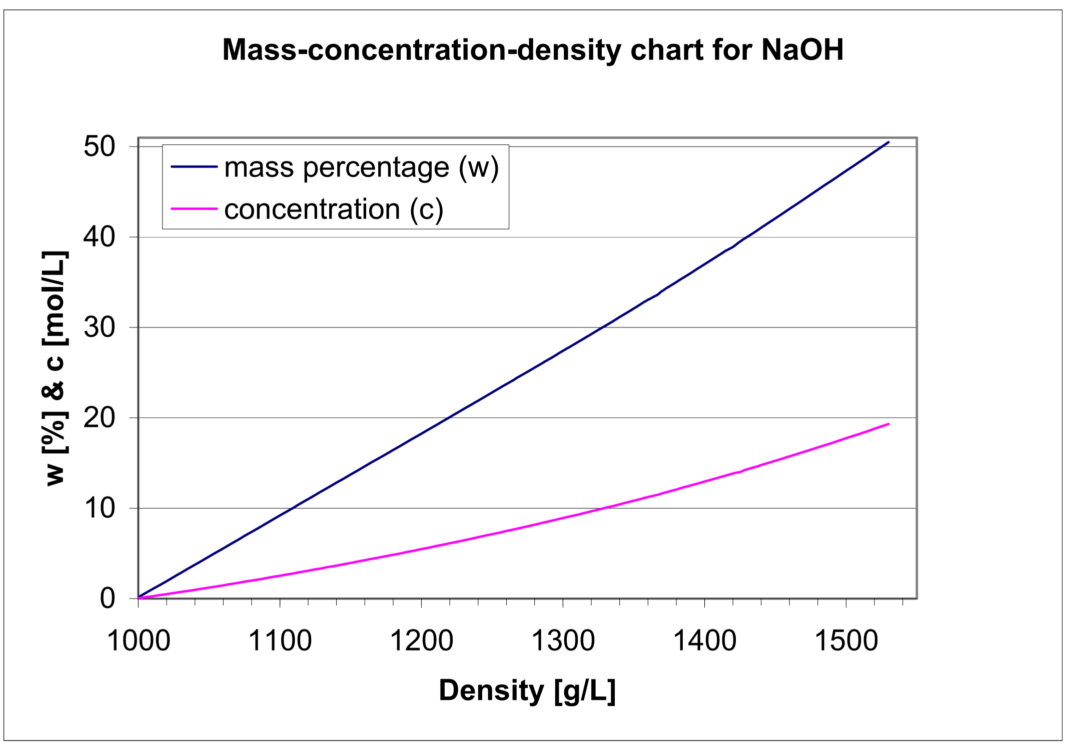
Mass-concentration-density chart for HCl:

density @20ºC	mass [%]	Conc. [mol/L]	density @20ºC	mass [%]	Conc. [mol/L]	density @20ºC	mass [%]	Conc. [mol/L]
1000	0.360	0.099	1070	14.945	4.253	1140	28.180	8.809
1005	1.360	0.375	1075	15.485	4.565	1145	29.170	9.159
1010	2.364	0.655	1080	16.470	4.878	1150	30.140	9.505
1015	3.374	0.939	1085	17.450	5.192	1155	31.140	9.863
1020	4.388	1.227	1090	18.430	5.510	1160	32.140	10.225
1025	5.408	1.520	1095	19.410	5.829	1165	33.160	10.595
1030	6.433	1.817	1100	20.390	6.150	1170	34.180	10.970
1035	7.464	2.118	1105	21.360	6.472	1175	35.200	11.340
1040	8.490	2.421	1110	22.330	6.796	1180	36.230	11.730
1045	9.510	2.725	1115	23.290	7.122	1185	37.270	12.110
1050	10.520	3.029	1120	24.250	7.449	1190	38.230	12.500
1055	11.520	3.333	1125	25.220	7.782	1195	39.370	12.900
1060	12.510	3.638	1130	26.200	8.118	1198	40.000	13.140
1065	13.500	3.944	1135	27.180	8.459			



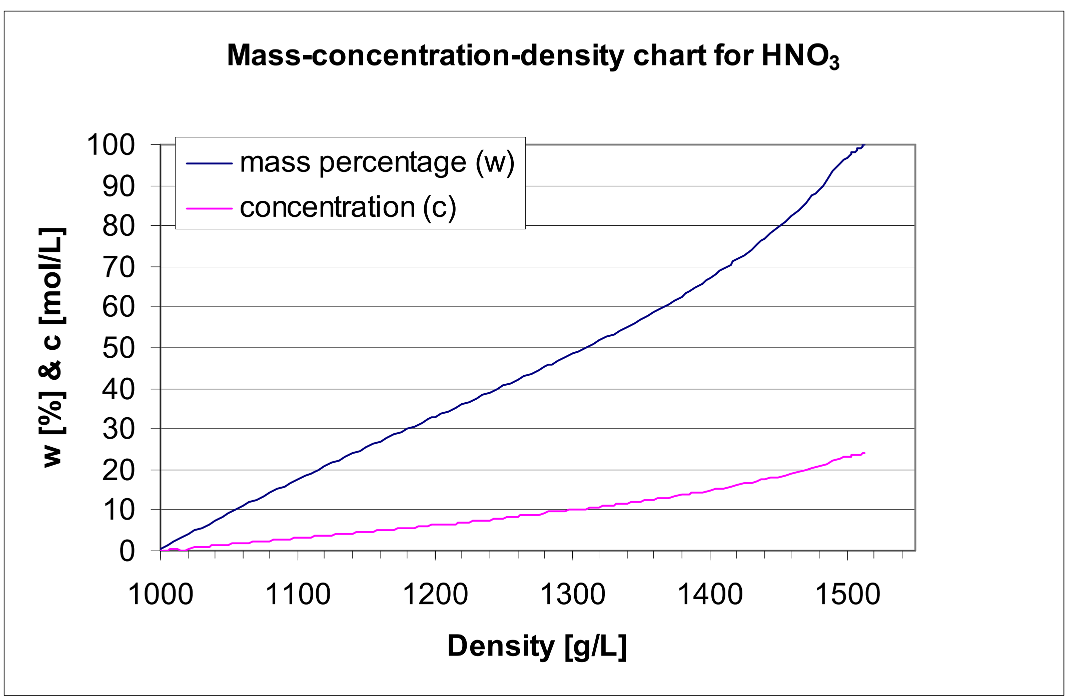
Mass-concentration-density chart for NaOH:

density @20ºC	mass [%]	Conc. [mol/L]	density @20ºC	mass [%]	Conc. [mol/L]	density @20ºC	mass [%]	Conc. [mol/L]
1000	0.150	0.038	1180	16.440	4.850	1360	33.060	11.240
1005	0.602	0.151	1185	16.890	5.004	1366	33.540	11.450
1010	1.045	0.264	1190	17.345	5.160	1370	34.030	11.650
1015	1.490	0.378	1195	17.800	5.317	1375	34.520	11.860
1020	1.940	0.494	1200	18.255	5.476	1380	35.010	12.080
1025	2.390	0.611	1205	18.710	5.636	1385	35.505	12.290
1030	2.840	0.731	1210	19.160	5.798	1390	36.000	12.510
1035	3.290	0.851	1215	19.620	5.958	1395	36.495	12.730
1040	3.745	0.971	1220	20.070	6.122	1400	36.990	12.950
1045	4.200	1.097	1225	20.530	6.286	1405	37.490	13.170
1050	4.655	1.222	1230	20.980	6.451	1410	37.990	13.390
1055	5.110	1.347	1235	21.440	6.619	1415	38.490	13.610
1060	5.560	1.474	1240	21.900	6.788	1420	38.900	13.840
1065	6.020	1.602	1245	22.360	6.958	1425	39.490	14.010
1070	6.470	1.731	1250	22.820	7.129	1430	40.000	14.300
1075	6.930	1.862	1255	23.275	7.302	1435	40.515	14.530
1080	7.380	1.992	1260	23.730	7.475	1440	41.030	14.770
1085	7.830	2.123	1265	24.190	7.650	1445	41.550	15.010
1090	8.280	2.257	1270	24.645	7.824	1450	42.070	15.250
1095	8.740	2.391	1275	25.100	8.000	1455	42.590	15.490
1100	9.190	2.527	1280	25.560	8.178	1460	43.120	15.740
1105	9.645	2.664	1285	26.020	8.367	1465	43.640	15.980
1110	10.100	2.802	1290	26.480	8.539	1470	44.170	16.230
1115	10.555	2.942	1295	26.940	8.722	1475	44.695	16.480
1120	11.010	3.082	1300	27.410	8.906	1480	45.220	16.730
1125	11.460	3.224	1305	27.870	9.092	1485	45.750	16.980
1130	11.920	3.367	1310	28.330	9.278	1490	46.270	17.230
1135	12.370	3.510	1315	28.800	9.466	1495	46.800	17.490
1140	12.830	3.655	1320	29.260	9.656	1500	47.330	17.750
1145	13.280	3.801	1325	29.730	9.847	1505	47.850	18.000
1150	13.730	3.947	1330	30.200	10.040	1510	48.380	18.260
1155	14.180	4.095	1335	30.670	10.230	1515	48.905	18.520
1160	14.640	4.244	1340	31.140	10.430	1520	49.440	18.780
1165	15.090	4.395	1345	31.620	10.630	1525	49.970	19.050
1170	15.540	4.545	1350	32.100	10.830	1530	50.500	19.310
1175	15.990	4.697	1355	32.580	11.030			



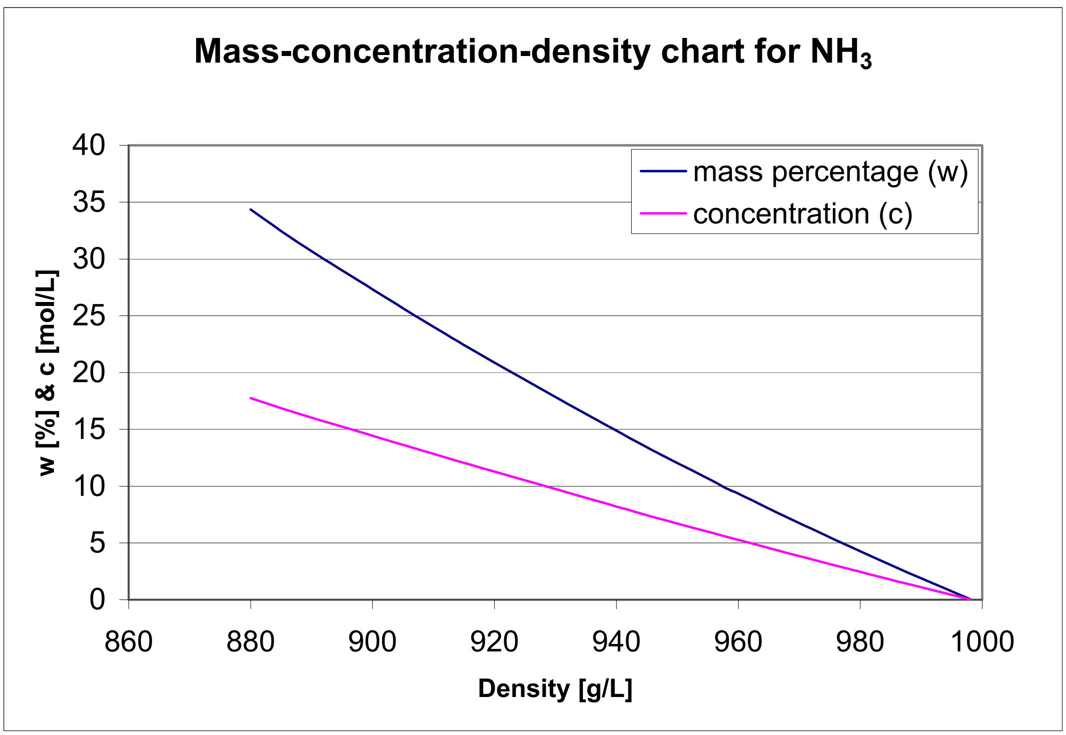
Mass-concentration-density chart for HNO_3:

density @20ºC	mass [%]	Conc. [mol/L]	density @20ºC	mass [%]	Conc. [mol/L]	density @20ºC	mass [%]	Conc. [mol/L]
1000	0.333	0.052	1190	31.470	5.943	1380	62.700	13.730
1005	1.255	0.200	1195	32.210	6.107	1385	63.720	14.010
1010	2.164	0.347	1200	32.940	6.273	1390	64.740	14.290
1015	3.073	0,495	1205	33.680	6.440	1395	65.840	14.570
1020	3.982	0,645	1210	34.410	6.607	1400	66.970	14.880
1025	4.883	0.794	1215	35.160	6.678	1405	68.100	15.180
1030	5.784	0.945	1220	35.930	6.956	1410	69.230	15.490
1035	6.661	1.094	1225	36.700	7.135	1415	70.390	15.810
1040	7.530	1.243	1230	37.480	7.315	1420	71.630	16.140
1045	8.398	1.393	1235	38.250	7.497	1425	72.860	16.470
1050	9.299	1.543	1240	39.020	7.619	1430	74.090	16.810
1055	10.121	1.694	1245	39.800	7.863	1435	75.350	17.160
1060	10.970	1.846	1250	40.580	8.099	1440	76.710	17.530
1065	11.810	1.997	1255	41.360	8.237	1445	78.070	17.900
1070	12.650	2.148	1260	42.140	8.426	1450	79.430	18.280
1075	13.480	2.301	1265	42.920	8.616	1455	80.880	18.680
1080	14.310	2.453	1270	43.700	8.808	1460	82.390	19.090
1085	15.130	2.605	1275	44.480	9.001	1465	83.910	19.510
1090	15.960	2.759	1280	45.270	9.195	1470	85.500	19.950
1095	16.760	2.913	1285	46.060	9.934	1475	87.290	20.430
1100	17.590	3.068	1290	46.850	9.590	1480	89.070	20.920
1105	18.390	3.224	1295	47.630	9.789	1485	91.130	21.480
1110	19.190	3.381	1300	48.420	9.990	1490	93.490	22.110
1115	20.000	3.539	1305	49.210	10.190	1495	95.460	22.650
1120	20.790	3.696	1310	50.000	10.390	1500	96.730	23.032
1125	21.590	3.854	1315	50.850	10.610	1501	96.990	23.100
1130	22.380	4.012	1320	51.710	10.830	1502	97.230	23.180
1135	23.160	4.171	1325	52.560	11.050	1503	97.490	23.250
1140	23.940	4.330	1330	53.410	11.270	1504	97.740	23.330
1145	24.710	4.489	1335	54.270	11.490	1505	97.990	23.400
1150	25.490	4.649	1340	55.130	11.720	1506	98.250	23.480
1155	26.240	4.810	1345	56.040	11.960	1507	98.500	23.560
1160	27.000	4.970	1350	56.950	12.200	1508	98.760	23.630
1165	27.760	5.132	1355	57.870	12.440	1509	99.010	23.710
1170	28.510	5.293	1360	58.780	12.690	1510	99.260	23.730
1175	29.250	5.455	1365	59.690	12.930	1511	99.520	23.860
1180	30.000	5.618	1370	60.670	13.190	1512	99.770	23.940
1185	30.740	5.780	1375	61.690	13.460	1513	100.000	24.010



Mass-concentration-density chart for NH_3:

density @20ºC	mass [%]	Conc. [mol/L]	density @20ºC	mass [%]	Conc. [mol/L]	density @20ºC	mass [%]	Conc. [mol/L]
998	0.047	0.027	958	9.810	5.550	918	21.500	11.590
996	0.512	0.299	956	10.400	5.840	916	22.125	11.900
994	0.977	0.570	954	10.950	6.130	914	22.750	12.210
992	1.430	0.834	952	11.490	6.420	912	23.390	12.520
990	1.890	1.100	950	12.030	6.710	910	24.030	12.840
988	2.350	1.365	948	12.580	7.000	908	24.680	13.160
986	2.820	1.635	946	13.140	7.290	906	25.330	13.480
984	3.300	1.910	944	13.710	7.600	904	26.000	13.800
982	3.780	2.180	942	14.290	7.910	902	26.670	14.120
980	4.270	2.460	940	14.880	8.210	900	27.330	14.440
978	4.760	2.730	938	15.470	8.520	898	28.000	14.760
976	5.250	3.010	936	16.060	8.830	896	28.670	15.080
974	5.750	3.290	934	16.650	9.130	894	29.330	15.400
972	6.250	3.570	932	17.240	9.440	892	30.000	15.710
970	6.750	3.840	930	17.850	9.750	890	30.685	16.040
968	7.260	4.120	928	18.450	10.060	888	31.370	16.360
966	7.770	4.410	926	19.060	10.370	886	32.090	16.690
964	8.290	4.690	924	19.670	10.670	884	32.840	17.050
962	8.820	4.980	922	20.270	10.970	882	33.595	17.400
960	9.340	5.270	920	20.880	11.280	880	34.350	17.750



Glossary:

Azeotropic Mixture: A liquid mixture whose boiling point is constant, so that the vapor pressure produced in distillation or partial evaporation has the same composition as the liquid phase. The boiling point of an azeotropic mixture will be at a minimum or maximum level compared to those of other mixture of the same substances.

Alcohol: An organic compound containing the hydroxyl group -OH.

Boiling Point: The temperature at which the vapor pressure of a liquid is equal to the external atmospheric pressure (KE_{Liquid} = KE of surrounding environment); the higher the intermolecular forces the lower the vapor pressure the higher is also the boiling point

Calorimeter: A device to measure the heat released/absorbed by a process under constant volume (isochorous) i.e.: $W = 0 \rightarrow \Delta U = Q$; the internal energy of a process will be converted into a change of heat.

Bomb C.: A combustion chamber w/n an isolated, sealed tank with stirrer, igniter and thermometer;

Calorimetry: The use of a calorimeter to measure the thermochemical properties of reactions.

Catalyst: A substance that increases the rate of a reaction without being consumed in the reaction; activation energy E_A is lowered significantly (increases rate of reaction) once catalyst is used in process without changing the overall energy of reaction (ΔH);

Complex Formation: A metal ligand coordinate-covalent bond formation.

Complex Ion: Ions containing a central metal cation bonded to one or more molecules or ions like the following sample show: $Ag(NH_3)_2^+$, $CdCl_4^{2-}$ $Cu(NH_3)_4^+$, $Fe(CN)_6^{3-}$, $Fe(H_2O)_6^{3+}$, $PtCl_4^-$, etc.

e.g.: $AgCl(s) + 2NH_3(aq) \leftrightarrow Ag(NH_3)_2^+(aq) + Cl^-(aq)$

according to Le Chattelier's principle, the removal of Ag^+ ions from the solution to form $Ag(NH_3)_2^+$ ions will cause more AgCl to dissolve, whereas if NH_3 is absent, more AgCl would precipitate.

Ligand: A group attached to a central metal ion in a complex, i.e.: molecules or ions that surround the metal atom and the ligands can be thought of a Lewis acid-base reaction; every ligand has at least one unshared pair of valence electrons (lone pairs), hence is a Lewis base; e.g.: H_2O, NH_3, CO, Cl^-, etc.

K_f CF. **Constant**: A measure of the tendency of a metal to form a particular complex ion; the larger K_f, the more stable the complex ion;

Compound: A substance composed of atoms of two or more elements chemically united in fixed properties.

Condensation: The phenomenon of going from gaseous state to the liquid state (see physics - matter).

C. **Reaction**: A reaction in which two smaller molecules combine to form a larger one. Water is invariably one of the products of such a reaction.

Crystal: An regular arrangement of atoms, ions, and molecules of periodically repeated, identically constituted, congruent lattice consisting of unit cells; see table below.

Crystalline Solid: A solid that possesses rigid and long-range order; its atoms, molecules, or ions occupy specific positions; e.g.: NaCl, diamond, graphite, etc.

Fractional C.: The separation of a mixture of substances into pure components on the basis of their differing solubility.

Crystallization: The process in which dissolved solute comes out of solution and forms crystals.

Recrystallization: Purification by repeated dissolving and crystallization.

Dalton's Law of partial P.: The partial pressure of a gas in a mixture is independent of other gases present; the total pressure is the sum of the partial pressure of all gasses present: x_A, mole fraction [-]

$p_A = x_A \cdot p_T$ [Pa] p_T, total pressure [Pa]

Deionized water: Water from which dissolved materials in the form of charged particles (ions) have been removed.

Distillation: The separation of a mixture by making use of the different volatilities of its components;

Fractional D.: A procedure for separating liquid components of a solution that is based on their different boiling points; based on Dalton's law of partial pressures;

see chemistry-gas: x_A, mole fraction [-]

$p_A = x_A \cdot p$ [Pa] p_T total pressure [Pa]

Distilled water: Water that has been boiled and recondensed to remove dissolved impurities; slow and expensive process.

Electrode: A metallic conductor that makes contact with an electrolyte in an electrochemical cell - see there.

Anode: (Gk: an, up) The electrode at which oxidation occurs; attracts anions; e.g.: Cl^-.

Cathode: (Gk: cat, down) The electrode at which reduction occurs; attracts cations; e.g.: Na^+.

SHE - **Standard Hydrogen** E.: A H-electrode that is in its standard state (H^+ ions at concentration 1[mol/l] and H-pressure 101[kPa]) and is defined as having $E° = 0$:

$H_2 \rightarrow 2H^+ + 2e^-$ $2H^+(aq, 1molar) + 2e^- \rightarrow H_2(g, 1atm)$ $E = 0$

Electrolyte: 1) An ionically conducting medium. 2) A substance that, when dissolved in water, results in a solution that can conduct electricity; see table below.

E. **Rule**: For a net potential of zero, the positive and negative charges must add up to zero; a solution must contain essentially as many anionic as cationic charges.

Non-E.: Is a solution in which no proportion of the solute molecules are ionized, hence does not conduct electricity; e.g.: $C_6H_{12}O_6(aq)$; see table below

Strong E.: Is a solution in which a large proportion of the solute molecules are ionized (complete dissociation into ions); 1mol $K_2SO_4 \rightarrow$ 3mol ions

e.g.: $NaCl(s) \rightarrow Na^+(aq) + Cl^-(aq)$ 1mol NaCl $\rightarrow$ 2mol ions

Weak E.: Is a solution in which only a small proportion of the solute molecules are ionized ionized (partly dissociation into ions); e.g.: $CH_3COOH(aq)$; see table below

H - **Enthalpy**: A thermodynamic quantity used to describe heat changes taking place at constant pressure (<u>isobar</u>); i.e.: reservoir of energy that can be obtained as heat;

$\Delta H = \Delta U + W = H_{final} - H_{initial}$ [kJ/mol]

$\Delta H = \Delta U + p\cdot\Delta V = \Delta U + R\cdot\Delta n\cdot T$ [kJ/mol]

$\Delta H < 0$: heat released (exothermic reaction);

$\Delta H > 0$: heat absorbed (endothermic reaction);

Reminder: when using ΔH, don't forget to add the reactants- and products phase!

ΔU, internal energy [J]
W, work [N·m] [J]
p, pressure [N/m^2] [Pa
ΔV, change in volume [m^3]
R, gas c. 8.314 [J/(K·mol)]
Δn, molar amount [mol]
T, temperature [K]

E. of **Chemical Change**: Processes involved in chemical changes;

- E. of **Reaction**: The difference between the enthalpies of the products and the enthalpies of the reactants; measured in [J/mole] (compare physics - heat).

  Endothermic R.: Processes that absorb heat from the surrounding environment, $\Delta H > 0$;

  i.e.: $\frac{1}{2} H_2(g) + \frac{1}{2} I_2(s) \rightarrow HI(g)$ $\Delta H = +25.9$ [kJ]

  Exothermic R.: Processes that give off heat to the surroundings, $\Delta H < 0$;

  i.e.: $H_2(g) + \frac{1}{2} O_2(g) \rightarrow H_2O(g)$ $\Delta H = -241.8$ [kJ]

Equation: An expression showing the chemical formulas of the reactants and products (both in symbols).

Ionic EQ.: An equation that shows dissolved ionic compounds in terms of their free ions;

i.e.: $Ag^+(aq) + NO_3^-(aq) + Na^+(aq) + Cl^-(aq) \rightarrow AgCl(s) + Na^+(aq) + NO_3^-(aq)$

Net Ionic EQ.: The equation showing the net change, obtained by canceling the spectator ions in an ionic equation; i.e.: $Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$

Writing ionic and net ionic EQ: 1) Write a balanced molecular EQ for the reaction; 2) Rewrite the equation to indicate which substance are in ionic form in solution (all electrolyte in solution dissociate into anions and cations; group-I elements); 3) Identify and cancel spectator ions (appear on both sides of the EQ) to arrive at the net ionic EQ;

Balanced EQ.: A chemical equation in which the same number of atoms of each element appear on both sides of the equation; i.e.: $2H_2 + O_2 \rightarrow 2H_2O$

Skeletal EQ.: An unbalanced equation that summarizes the qualitative information about the reaction;

i.e.: $H_2 + O_2, \rightarrow H_2O,$

Product: A substance formed in a chemical equation.

Reactant: A starting material in a chemical reaction; a reagent taking part in a specified reaction.

Symbol: One- or two-letter abbreviation of an element's name.

Equilibrium: The state of final balance of a multi-compound homogenous mixture;

for K_C, Q_C, Dynamic E. etc.

K_C - E. **Constant**: A number equal to the ratio of the equilibrium concentration of <u>gaseous</u> products to the equilibrium concentrations of <u>gaseous</u> reactants, each raised to the power of its stoichiometric coefficient (ignored when in their solid or liquid state); by convention, numerator stands for products, and denominator stands for reactants; K_C is <u>temperature dependent</u>;

any solid that precipitate or compound that liquefies, is left out.

e.g.: $aA + bB \leftrightarrow cC + dD$ k_F, k_R, rate c. of forward /reverse reactions [mol/(l·s)]

$K_C = k_F/k_R = c^c_{(C)}\cdot c^d_{(D)} / (c^a_{(A)}\cdot c^b_{(B)})$ [var]

$K_C > 10^3$: favors products strongly

$K_C > 10^{-3}$-10^3: reactants & products at equilibrium

$K_C < 10^{-3}$: favors reactants strongly

Dynamic E.: The condition in which a foreword process and its reverse are occurring simultaneously at equal rates; e.g.: vaporizing and condensing; chemical reactions at equilibrium, etc.; e.g.:

$H_2(g) + I_2(g) \leftrightarrow 2HI(g)$;

$v_F = k_F\cdot c_{(H2)}\cdot c_{(I2)}$; $v_R = k_R\cdot c^2_{(HI)}$;

$v_F = v_R$ in equilibrium: $c^2_{(HI)}/(c_{(H2)}\cdot c_{(I2)}) = k_F/k_R = K$

v_F, v_R, rate of forward/reverse
k_F, k_R, rate constants [1/s]
$c_{(x)}$, molar concentration [mol/l]

Esters: Compounds that have the general formula R'COOR; R' can be H or an alkyl group or an aryl group.

Q - **Heat**: The amount of energy in form of heat;

$Q = \Delta H \cdot n = C \cdot \Delta T$ [kJ]

C - **Heat Capacity**: The amount of heat required to raise the temperature of a given quantity of the substance by 1°C; i.e.: to raise 1g of water from 14.5°C to 15.5°C the energy of 4.184[J] (=1cal) is required: $C = m \cdot s$ [J/K]

n, molar amount	[mol]
C, heat capacity	[J/K]
T, temperature	[K]
m, mass	[g]
s, specific heat	[J/(g·K)]

H. of **Dilution**: The heat change associated with the hydration process - see chemistry liquid.

H. of **Hydration**: The heat change associated with the hydration process.

H. of **Solution**: see enthalpy of solution.

Specific H. **Capacity**: The heat capacity per gram.

$s = Q/(m \cdot \Delta T)$ [N·m/(kg·K)] = [J/(kg·K)]

Q, heat [N·m]	[J]
m, mass	[kg
T, temperature	[K]

Hydrates: Compounds that have a specific number of water molecules attached to them; e.g.: $CuSO_4 \cdot 5H_2O$;

Hydration: A process in which an ion or a molecule is surrounded by water molecules arranged in a specific manner; e.g.: water - H_2O molecules attach to a central ion (ion-dipole interaction).

Hydrated Anion: Hydrogen bonds form between the H of water and the central anion; e.g.: SO_4^{2-}.

Hydrated Cation: Ion-dipole forces between the O of water and the central ion are responsible; e.g.: Be^{2+}.

H. **Crystals**: Hydrated ions remain intact even in a solidified structure;

e.g.: $[Fe(OH_2)_6]^{3+}\ Cl_3^{3-}$ actual structure — notation: $Fe_2Cl_3 \cdot 6H_2O$

or $[Cu(OH_2)_4]^{2+}\ [SO_4(H_2O)]^{2-}$ — notation: $CuSO_4 \cdot 5H_2O$

Hydrophillic: Water-liking.

Hydrophobic: Water-fearing.

Indicator: A weak organic acid or base that change color when it goes from its acid to its base form; i.e. acid-base neutralization (an acid-base indicator) or from its oxidized to its reduced form (a redox indicator); e.g.:

$HInd(aq) + H_2O(l) \leftrightarrow H_3O^+(aq) + Ind^-(aq)$ where $c_{(H3O+)}$ is the H^+ concentration (pH) of the anylate.

$K_{(Ind)} / c_{(H3O+)} = c_{(HInd)} / c_{(Ind-)}$ [mol/l]

if = $c_{(HInd)} / c_{(Ind-)} > 1$, color of acid (Hind) predominates

if = $c_{(HInd)} / c_{(In-)} < 1$, color of conjugate base (Ind^-) predominates

Preconditions are: both 1) HIn and In^- have to be water soluble, 2) HIn and In^- have to separate colors, 3) Indicator concentration $c_{(HIn)}$ has to be low, 4) $c_{(H3O+)}$ of indicator should measure only pH of solution.

Note: More than two drops of indicator would upset the accuracy of the titration;

Ion: An atom or molecule that has lost or gained one or more electrons, and thus becomes positively or negatively charged; i.e.: Al^{3+} (mono-atomic ion), SO_4^- (poly-atomic ion); see also chemistry-atom.

Anion: An ion with a net negative charge, i.e.: F^-, SO_4^-, etc; see table below.

Cation: An ion with a net positive charge, i.e.: Na^+, NH_4^+, Al^{3+}, etc; see table below.

Ionic Compound: Any neutral compound containing cations and anions.

Ion Pair: A species made up of at least 1 cation and at least 1 anion held together by electrostatic forces.

Ionization: Conversion to cations by the removal of electrons; see chemistry-thermochemistry ΔH.

e.g.: $K(g) \rightarrow K^+(g) + e^-(g)$

Polarized I.: The distorted electron cloud of an ion (or atom); see also chemistry atom; e.g.:

Anion (>cloud; >charge): I^- (r = 220pm) easier distortable than F^- (r = 133pm); S^{2-} easier polarizable than Cl^-.

Cation (<cloud; <charge): Li^{2+} more covalent than Cs^+; Be^{2+} more covalent then cations of 4th period.

Law of Mass Action: For an equilibrium of the form $aA + bB \leftrightarrow cC + dD$, the reaction quotient $Q_C = c^c_{(C)} \cdot c^d_{(D)} / c^a_{(A)} \cdot c^b_{(B)}$; evaluated by using the equilibrium molar concentrations of the reactants and products, is equal to a constant K_C which has a specific value for a given reaction and temperature.

Mass Units: The following equations are commonly used in dealing with masses in chemical equations:

β - **Mass Concentration**: The mass of solute per liter of solution: $m_{(x)}$, mass [g]
$\beta_{(x)} = m_{(x)} / V_{(Sln)}$ [g/l] $V_{(Sln)}$, volume of solution [l]
ρ - **Density**: The mass of a substance divided by its volume $m_{(Sln)}$, mass [g]
$\rho_{(Sln)} = m_{(Sln)} / V_{(Sln)}$ [g/l] $V_{(Sln)}$, volume of solution [l]
M_r **- Molecular** M.: The sum of the atomic masses (in amu) present in the molecule;
i.e.: $M_r(H_2O) = 2\cdot1.008$ (atomic mass of H) + 16.00 (atomic mass) of O = 18.02 [amu].
n - **Molar Amount**: The amount of an element per molar mass: $m_{(x)}$, mass [g]
$n_{(x)} = m_{(x)} / M_{(x)}$ [mol] $M_{(x)}$, molar mass [g/mol]
M - **Molar Mass**: The relative mass (g, or kg) per mole of atoms (amu) , molecules, or other particles - see also atom-mass: m_{av}, average mass [amu]
$M_{(x)} = m_{av}\cdot N_A$ [g/mol] N_A, Avogadro's c.$6.022\cdot10^{23}$ [atoms/mol]
i.e.: $M(H_2O) = 2\cdot1.008$ [g/mol]of H + 16.00 [g/mol] of O = 18.02 [g/mol];
Molar Mass Fraction: Ratio of the number of moles of one component of a mixture to the total number of moles of all components in the mixture; i.e.: the respective mass of H and O in a given sample of water is obtained by:
$m_H = m_{H2O} \cdot 2\cdot M_H/M_{H2O}$ [g]; $m_O = m_{H2O} \cdot M_O / M_{H2O}$ [g];

Molar: The quantity per mole; i.e.: molar mass (the mass per mole), molar volume (the volume per mole), etc.
b - **Molality**: see chemistry-liquids.
c - **Molar Concentration**: see chemistry-liquid.
Molar Solubility: see chemistry-liquid.
c - **Molarity**: see chemistry-liquid.

Mole: (L, massive, heap) The SI base unit for the amount of substances that contains as many elementary entities (atoms, molecules, or other particles) as there are atoms in exactly 12 grams of the carbon-12 isotope; always equal to Avogadro's number = $6.02205\cdot10^{23}$.

Nernst Equation: The EQ expressing the cell potential in terms of the concentrations of the reagents taking part in the cell reaction; K_C, equilibrium constant [var]
$E°$, stand. reduction pot. [V]
$E = E° - \ln(q)\cdot R\cdot T/(N_{OX}\cdot F) = E° - \log(K_C)\cdot0.05916/N_{OX}$ [V] R, gas constant 8314 [J/mol]
e.g.: $Zn(s) + Cu^{2+}(aq) \rightarrow Zn^{2+}(aq) + Cu$ (s) T, temperature [K]
$= 1.1\text{-}\log(c_{(Zn2+)}/c_{(Cu2+)})\cdot0.0592/2$ N_{OX}, oxidation number [-]
Redoxpotential of any half reaction of a metal M^{n+}, molar concentration on metal ion in solution [mol/l]
$E = E° + \log(M^{n+})\cdot0.05916/N_{OX}$ [V]

Meniscus: The curved surface of a liquid in a narrow glass tube (buret, pipet, etc.); see also appendix-utensils.

Mixture: A type of matter that consists of more than one substance and may be separated into components by making use of the different physical properties.
Heterogeneous M.: A mixture in which the individual components, although mixed together, lie in distinct regions, even on a microscopic scale; e.g.: a mixture of sand and sugar, ect.
Homogenous M.: A mixture in which the individual components are uniformly mixed, even on an atomic scale; e.g.: air, solutions, etc.

Molecule: 1) Smallest possible unit of a compound, that possesses the chemical properties of the compound. 2) A definite and distinct, electrically neutral group of bonded atoms; i.e.: H_2, NH_3, CH_3COOH, etc.

Oxidation: 1) Combination with oxygen. 2) A reaction in which an atom, ion, or molecule loses an electron;
e.g.: $Ca(s) \rightarrow Ca^{2+}(s) + 2e^-$ (represents the Ca in CaO).
Oxidation corresponds to an increase of oxidation number.
O. **Agent**: A substance that can accept electrons from another substance or increase the oxidation number in another substance (being oxidized); where the substance itself is reduced;
e.g.: O_2, O_3, MnO_4^-, Fe^{3+}
N_{ox} - O. **Number**: The effective charge on an atom in a compound, calculated according to a set of rules. An increase in ON. corresponds to oxidation, and a decrease to reduction.

RedOx Reaction: A reaction in which there is either a transfer of electrons or a change in the oxidation numbers of the substances taking part in the reaction. Oxidation and reduction takes place simultaneously, because an electron that is lost by one atom is accepted by another. Oxidation-reduction reactions are important means of energy transfer in living systems. e.g.: $Ca(s) + ½O_2(g) \rightarrow CaO(s)$;

Reduction: (L. reductio, bringing back) 1) The removal of oxygen form (bringing back a metal from its oxide) or the addition of hydrogen to a compound. 2) A reaction in which an atom, an ion, or a molecule gains an electron; reduction takes place simultaneously with oxidation;
e.g.: $½O_2(g) + 2e^- \rightarrow O^{2-}$ (represents the O in CaO).
Reduction corresponds to a decrease in oxidation number.

Salt: An ionic compound made up of a cation other than H^+ and an anion other than OH^- or O^{2-}.

S. **Hydrolysis**: The reaction of the anion or cation, or both, of a salt with water.

Significance:

Significant Figures: The number of meaningful digits in a measured or calculated quantity.

Solubility: The maximum amount of solute that can be dissolved in a given quantity of a specific solvent at a specific temperature (for gases: at a specific pressure); the concentration of a saturated solution of a substance; e.g.: how much of a salt can be dissolved in a solvent.

K_S - S. **Constant**: see solubility product;

K_{sp} - S. **Product**: The product of relative ionic molar concentrations of the constituent ions in a saturated solution, each raised to the power of its stoichiometric coefficient in the equilibrium EQ;

c, ionic molar conctr. [mol/l]
A^-, C^+, anion, cation [-]
K_C, equilibrium constant [-]

$K_{sp} = K_C \cdot c_{(HA)} = c_{(A-)} \cdot c_{(C+)}$ [mol^2/l^2]

e.g.: $Hg_2Cl_2(s) \leftrightarrow Hg_2^{2+}(aq) + 2Cl^-(aq)$; $K_{sp} = c_{(Hg2++)} \cdot c^2_{(Cl-)}$ [mol^3/l^3]

Q_{sp} - S. **Quotient**: The molar analogue of the solubility product, but with the molar concentrations not necessarily those at equilibrium.

$Q_{sp} \geq K_{sp}$ precipitate will form, whereas if $Q_{sp} < K_{sp,}$ still more salt can be added and will dissolve.

S. **Rules**: Solubility pattern of a range of common compounds in water - see table below.

- unpolar and polar substances are not miscible; e.g.: oil and water.
- like dissolves like; e.g.: ionic bonded element dissolve well in polar solvents, NaCl in H_2O.

Sln - **Solution**: A homogeneous mixture of two or more substances.

Aqueous S.: A solution in which the solvent is water.

Enthalpy of S.: see chemistry - thermochemistry.

S. **Concentration**: The amount of solute present in a given quantity of solution.

Ideal S.: Any solution that obeys Raoult's law at any concentrations. Real solutions resembles ideal solutions more closely the lower the concentration; below 0.1[mol/kg] for non electrolytic solutions and 0.01[mol/kg] for electrolytic solutions.

Dilution: A procedure for preparing a less concentrated solution from a more concentrated solution; Reminder: $n_{(concentrades\ solvent)} = n_{(diluted\ solvent)}$;

If a solute is added in very small quantities compared to the solvent, than the vapor pressure of the liquid can be said to be equal to that of the pure solvent; any diluted liquid given as a %-value usually refers to mass-% in a 100g of solution;

being so diluted Raoult's law can be implemented:
molar fraction of solute B = $x_{(B)} = \Delta p / p°_{(A)}$ [-]

Δp, change of vapor pressure of solution
$p°_{(A)}$, vapor pressure of pure solvent [Pa]

Saturated S.: At a given temperature, the solution that results when the maximum amount of a substance has dissolved in a solvent; dissolved and undissolved solute are in dynamic equilibrium.

- **Oversaturated** S.: (supersaturated) A solution that contains more solute than it has the capacity to dissolve (unstable).
- **Unsaturated** S.: A solution that contains less solute than it has the capacity to dissolve.

Standard S.: A solution of accurately known concentration, used for acid-base tritations to calculate the dosage of molar amounts out of a used volume; see chemistry-acid-base.

Slt - **Solvent**: The substance (usually one, or more) present in larger amount in a solution.

Sle - **Solute**: The substance present in smaller amount in a solution.

Stoichiometry: The mass relationships among reactants and products in chemical reactions.

S. **Amount**: The exact molar amount of reactants and products that appear in a balanced chemical EQ.

S. **Coefficient**: The number of moles of each substance in a chemical equation; i.e.: 1 and 2 in: $H_2 + Br_2 \rightarrow 2HBr$

S. **Point**: The stage in a titration when exactly the right volume of solution needed to complete the reaction has been added (see chemistry - acid and base).

S. **Proportions**: Reactants in the same proportions as their coefficients in the chemical equation; i.e.: equal amounts of H_2 and Br_2 in the reaction mentioned above.

S. **Relation**: An expression that equates the relative amounts of reactants and products that participate in a reaction; i.e.: 1 mol H_2 = 2 mol HBr.

Reaction S.: The quantitative relation between the amounts of reactants consumed and products formed in chemical reactions as expressed by the balanced chemical equation for the reaction.

Titration: The analysis of composition by measuring the volume of one solution (the titrant) needed to react with a given volume of another solution (the anylate).

Anylate: The solution of unknown concentration in a titration.

Titrant: The solution of known concentration added from a buret in a titration.

Temperature: How hot or cold a sample is; the intensive property that determines the direction in which heat will flow between two objects in contact.

Titer: The reacting strength or concentration of a solution as determined by titration with a standard.

V - **Potential** (voltage): The electric (pressure) potential energy per amount of charge, measured in volts, see physics electromagnetics.

PE, potential energy [J]
q, charge [A·s] [C]

$V = PE/q$ [J/(A·s)] = [J/C] = [V]

Vapor: The gaseous phase of a substance (specifically, of a substance that is a liquid or a solid at the temperature in question); see chemistry - liquid.

V. **Pressure**: The pressure exerted by the vapor of a liquid (or solid) when the vapor and the liquid (or solid) are in dynamic equilibrium; the higher the intermolecular forces the lower the vapor pressure; e.g.: $H_2O(g) \leftrightarrow H_2O(l)$

Volatility: The readiness with which a substance vaporizes: A substance is typically regarded as volatile if it its boiling point is below 100°C.

Volatile: as a measurable vapor pressure.

Yield: The outcome of a chemical reaction, expressed in grams, mole, liters, etc.

Y. of **Reaction** (actual yield): The quantity of product obtained from the reaction.

Percentage Y.: The percentage of the theoretical yield of a product achieved in practice

$Y_{\%} = 100 \cdot Y_A / Y_T$ [-]:

Theoretical Y.: The amount of product predicted by the balanced equation when all of the limiting reagent has reacted.

Y_A, achieved yield [g]
Y_T, theoretical yield [g]

Limiting Reagent: The reactant that governs the theoretical yield of product in a given reaction.

HP41 Software Routine to rapidly calculate the pH (Quadratic Equation; Exp. 7):

The following software routine written for the Hewlett Packard 41CVX-Pocket Computers yields quick access to the theoretical pH of the buffer system. For this purpose it is essential to know the following parameters to allow the subsequent calculations to be made:

$$_1x_2 = \frac{-b \pm \sqrt{b^2 - 4 \cdot a \cdot c}}{2 \cdot a}$$

- $_1x_2$ are the two alternative solution of this equation
- a, b, c, are the constants for the general expression of the quadratic equation: $a \cdot x^2 + b \cdot x + c = 0$

Software written by P. Madl for the HP-41CV

Step	Command	Brief description	Status
1	**LBL QEQ**		
2	SF 00	Set PRGM-running flag.	SF 00
3	CF 27		
4	‘A = ?	Enter the value of the 1st constant (a) and store in memory locus 10.	
5	PROMPT		STO 10
6	STO 10		
7	‘B = ?	Enter the value of the 1st constant (b) and store in memory locus 11.	
8	PROMPT		STO 11
9	STO 11		
10	‘C = ?	Enter the value of the 1st constant (c) and store in memory locus 12.	
11	PROMPT		STO 12
12	STO 12		
13	XEQ 01	Go to subroutine 01 which calculates the nominator and come back	
14	-	Calculation of denominator for X1	
15	RCL 10s		
16	2		
17	*		
18	/		
19	‘X1 =	Display 1st solution of quadratic equation	
20	XEQ 04	Execute subroutine 04 (subroutine used to display results)	
21	FC? 01	If results are in the complex regime, continue with subroutine 6	
22	**GTO 06**		
23	RCL 13		
24	X≤0?		
25	XEQ 05	Execute subroutine 5 (subroutine used to check for imaginary results of the QEQ)	
26	**LBL 06**	Subroutine to calculate solution X2	
27	XEQ 01	Go to subroutine 01 which calculates the nominator and come back	
28	+	Calculation of denominator for X2	
29	RCL 10		
30	2		
31	*		
32	/		
33	‘X2 =	Display 1st solution of quadratic equation	
34	FC? 01	Check flag (complex numbers) – start calculation of imaginary results	
35	CF 00		
36	CF 01	Clear Flag that indicates complex number results	
37	FC? 00	If program flag 00 is not set, terminate calculation and go to label 4	
38	**GTO 04**		
39	XEQ 04	Execute subroutine 04 (subroutine used to display results)	
40	RCL 13		
41	CHS		
42	X ≤ 0?		
43	FC? 01		
44	**GTO 05**	Execute subroutine 05 (subroutine used to check for imaginary results)	
45	XEQ 05	Execute subroutine 05 (subroutine used to check for imaginary results)	
46	RTN		
47	**LBL 01**	Subroutine to calculate the real components of the QEQ	
48	RCL 11		
49	CHS		
50	RCL 11		
51	X^2		
52	RCL 10		
53	RCL 12		
54	*		
55	4		

Step	Command	Brief description	Status
56	*		
57	-		
58	X < 0?		
59	**GTO 02**	Execute subroutine 02 (subroutine used to calculate imaginary components)	
60	SQRT		
61	**LBL 03**		
62	RTN		
63	**LBL 02**	Subroutine for the calculation of imaginary results (complex numbers)	
64	SF 01	Set Flag 01 to indicate that results are in the complex regime	SF 01
65	‘COMPLEX!	Indicate Complex number regime by prompting COMPLEX!	
66	PROMPT		
67	CHS		
68	SQRT		
69	RCL 10		
70	2		
71	*		
72	/		
73	STO 13	Memory allocation of complex number results in memory 13	STO 13
74	RCL 11		
75	CHS		
76	0		
77	GTO 03		
78	**LBL 05**	Subroutine for displaying imaginary results (complex numbers)	
79	‘IM =		
80	FC? 01		
81	CF 00		
82	FC? 00		
83	**GTO 04**		
84	XEQ 04		
85	RTN		
86	**LBL 04**	Subroutine for displaying real results (real values)	
87	ARCL X		
88	AVIEW		
89	FS? 00		
90	STOP		
91	FS? 00	If program flag 00 is still set return to main menu otherwise exit program.	
92	RTN		
93	END		
94			