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Medical chemistry

04.09.12

http://tera.chem.ut.ee/~koit/arstpr/intro_en.pdf

1 Introduction

Medicinal chemistry is a chemistry-based discipline, also involving aspects of biological, medical and pharmaceutical sciences. It is concerned with the invention, discovery, design, identification and preparation of biologically active compounds, the study of their metabolism, the interpretation of their mode of action at the molecular level and the construction of structure-activity relationships.

(Glossary Of Terms Used In Medicinal Chemistry (IUPAC Recommendations 1998);

<http://www.chem.qmul.ac.uk/iupac/medchem/>)

2 Analytical medical chemistry

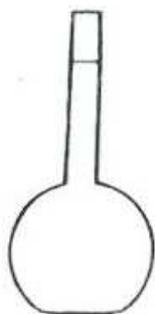
Analytical chemistry is a part of chemistry that studies composition of substances. Analytical medical chemistry deals with medical and biological samples.

Qualitative analysis gives as a result the list of compounds that are present in the mixture of compounds.

Quantitative analysis allows the determination of compound(s) amount in the mixture of compounds. In current lab you will learn some analytical chemistry methods that are used in medicine.

3 Common laboratory glassware

3.1 Volumetric flask



Volumetric flask is meant for preparing known amount of solution with necessary concentration. The volume of volumetric flask is marked on the flask's neck or side. Flasks are prepared for volumes from 1 ml to few litres. Most common are flasks for 50 and 100 ml.

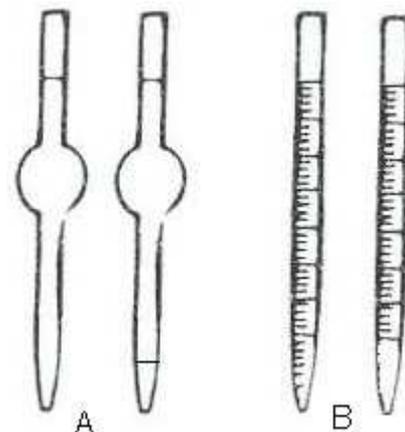
3.2 Pipette

Pipettes are used for measuring known volume of liquid. The (maximum) amount measured with the pipette is marked on the upper part of the pipette.

Two types of pipettes are used:

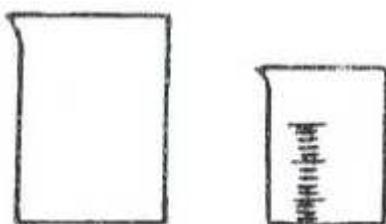
Volumetric pipette (figure A) – for measuring one certain volume. Eg. If marked volume on the pipette is 10 ml, then this is the only volume that can be measured accurately with that pipette.

Graduated pipette (figure B) – maximum volume of liquid measurable is the volume marked on the pipette. Eg. If graduated pipette is marked with volume 10 ml, then it allows measuring 0 – 10 ml of liquid.



Pasteur pipette (also called drop-pipet) is not used for measuring, it is used for filling volumetric flask to the mark.

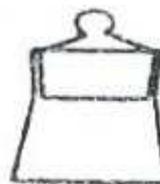
3.3 Beaker



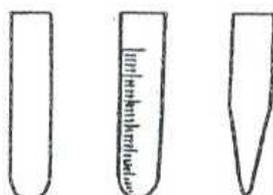
Vessel for pouring out or temporary storage of liquids. Even though sometimes beaker does have volume markings it is not calibrated measure!

3.4 Weighing bottle

Vessel that can be closed with a lid and is used for weighing substance.



3.5 Test-tubes

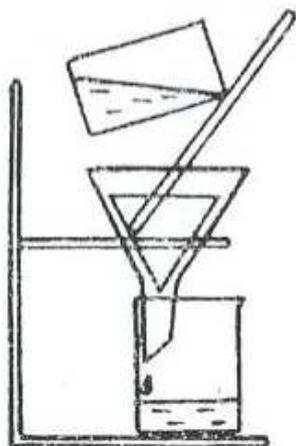


Vessels for carrying out reactions.

Centrifuge glass tube – conically shaped (test-tube that is thinner from the bottom).

Test-tubes are not calibrated measures even if volumes are marked on it. However, sometimes they are used for measuring approximate volumes.

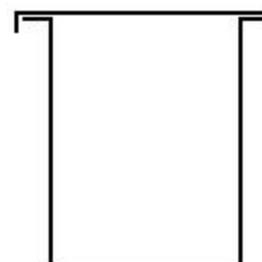
3.6 Filtering device



Is used for separating solid residue from the liquid phase. Very commonly fluted filter of paper is used.

3.7 Solvent chamber

Vessel that can be covered with a lid and is used in thin layer chromatography.



4 Working with the laboratory glassware

4.1 Washing glassware

Before using glassware they must be washed three times with distilled water. Wash-bottle filled with distilled water is used for washing. For washing vessels small amount of distilled water is poured into the bottom of the vessel and then poured out. This step is repeated three times.

If a prepared solution is planned to be poured into the vessel, the vessel must be washed three times with the solution as described above.

If a solution is planned to be prepared by dilution, the vessel must be washed only with distilled water. After washing the vessel it should not be touched from the inside with dirty (unwashed) objects or hands.

4.2 Instruction for using pipette filler

Pipette filler is shown on the figure.

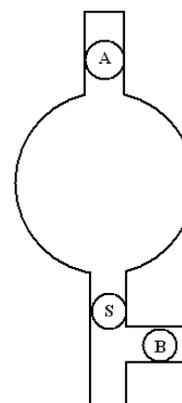
To empty pipette filler, spot A is squeezed and at the same time the ball is pressed to let some of the air out.

Pipette is attached to the bottom part of the pipette filler.

In order to fill the pipette with the liquid, spot S is pressed.

In order to empty the pipette B is pressed.

Avoid liquid from entering the pipette filler!



pipette filler

4.3 Washing pipettes

All pipettes are also washed three times with distilled water. While washing pipette it should be filled with washing liquid to more than the last measuring mark since when filling a pipette with a solution it will be first filled more than the last measuring mark. It is important that while washing the larger area is washed (a) than what is used later for filling a pipette (b). The next step is drying the pipette outside with the paper that is followed by washing the pipette with the solution that is being measured. For filling pipettes concentrated solution is never taken straight from the vessel where it is prepared in but it is necessary to pour it out into the beaker.

4.4 Pipetting

Pipette is always hold vertical while filling or emptying. Emptying the pipette is carried out so that the tip of the pipette is situated against the wall of the vessel where the pipette is emptied into (it is also allowed to „lean“ the tip on the surface of the liquid). This is done in order to avoid splashing and allow the continuous flowing of the liquid out of the pipette. Pipette must never be emptied into the vessel where it was taken from. The excess liquid must be emptied into a beaker or to a waste where all excess liquid from washing are collected.

4.5 Filling and emptying of the pipette

Pipettes are meant for measuring and moving solutions of known volume from one vessel to another. Pipette's volume is marked on the neck of the pipette. In order to get the necessary amount of solution into a pipette, it is filled over the mark (figure b) and then the excess of the solution that is above the mark is emptied into the waste. Close attention is to be kept on the mark so that the lowest part of the liquid level (meniscus) is leveled with the mark (figure c). In order to observe, the mark must be on the same height as the eye (d).

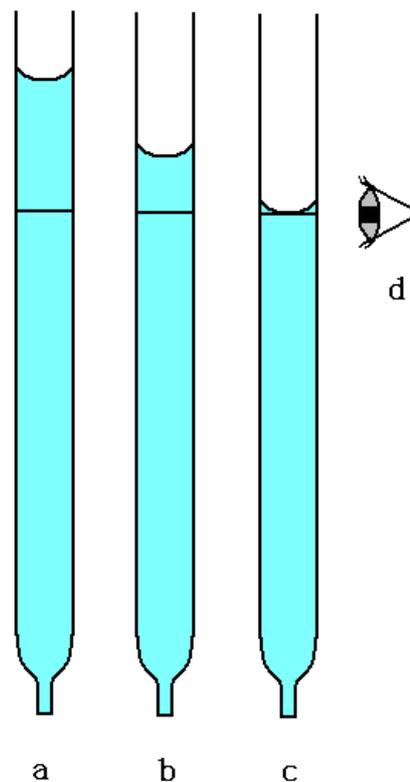
If the pipette has only one mark (volumetric pipette), it must be emptied up to the end in order to get the accurate volume (pipette must be hold vertically and against the wall of the vessel above the liquid level). This way pipette empties the right amount, leaving a small amount of liquid in the tip of the pipette. In no case this liquid should be emptied into the vessel where the liquid is measured into.

When emptying pipettes with the scale (graduated pipettes) it should be emptied by fraction and the liquid level should be leveled with the mark as described above (c, d). For some graduated pipettes the last part of the scale is on the tip of the pipette, then the emptying is as for volumetric pipettes. If the scale of the graduated pipette is above the tip, the maximum volume for measuring is the difference between the highest and the lowest mark. This kind of graduated pipette should never be emptied up to the end but until the last mark.

4.6 Preparing the solution into the volumetric flask

Preparation of the solution into the volumetric flask is done by dilution. First, the needed amount of the substance or its solution is analytically carried into the volumetric flask. Analytical carrying means that all of the needed amount of substance is carried over.

If the substance is solid, the vessel where it was kept before, must be washed with small amounts of the solvent (until the vessel is clean) and this must be added into the volumetric flask. If glassfunnel and glass-rod or spatula were used, these must be washed as well so that all the washing solvent ends up in the volumetric flask.

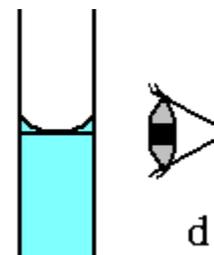


If the solution of the substance is added with the pipette, it must be kept in attention that the required amount of the solution is added. The tip of the pipette must be below the mark (and pushed against the wall of the volumetric flask).

After the analytical carrying of the substance, the volumetric flask is filled with the distilled water (or other solvent) almost to the mark on the wall of the flask. The last part of the flask is filled to the mark with the drop-pipet.

Flask is filled correctly if the mark is leveled with the bottom part of the liquid level. For the observation, the mark must be the same height as the eye (d).

After the filling of the flask, the solution must be mixed. For that, the flask is closed with the stopper and the hand is placed around the neck of the flask so that the finger is above the lid and the flask is turned 180° so that the stopper is pointed downwards. It is waited until all the bubbles have moved to the bottom of the flask and then the flask is turned up again. This should be repeated at least ten times to ensure full mixing.



5 Finishing work

After the work, all glassware must be washed (3 times with tap water and 3 times with distilled water). Workspace must be tidied up also.

6 Using analytical scales

Analytical scales are meant for weighing small amounts of substances. Next to the keyboard of the scale, the maximum mass which is allowed to weigh with that scale is marked (in this case 180 grams). If the weighed object is heavier than the maximum mass, it must be immediately removed from the scale. Before using the scale, ensure that the balance compartment is clean. If necessary, clean it gently with the feather.

Close all the glass doors of the scale.

In order to turn off the scale, switch ON/OFF button.

To move all the objects onto the scale, use tweezers.

All solid weighed portions must be weighed in weighing bottle.

For weighing, the glass door is opened and using the tweezers the empty weighing bottle is moved inside the balance compartment.

Weighing bottle is put in the centre of the balance.

Glass door is closed and it is waited until the reading stabilizes.

Then the reading is tared (zeroed) using the button TARE.

It is waited until the reading is 0.0000 g.

Glass door is opened and the weighing bottle is taken out using tweezers.

It is not allowed to add substance into a weighing bottle when it is on the balance.

Necessary amount of the substance is added into the weighing bottle. If the necessary amount is not known, the adding of the substance starts by small portions and weighing after every addition until the necessary amount is obtained.

After adding, the weighing bottle is taken onto the balance and the glass door is closed.

After the stabilization of the reading, it must be recorded at all times as well as what was the weighed object (substance, sample nr, prepared solution nr) and also the mass units (in this case grams).

If necessary, the reading of the scale could be tared again and then the next substance could be added (outside the scale).

7 Tasks for the first lab

Team up in 3-5 persons groups. Record the results (one protocol per group).

1. Practice filling a pipette to the mark using pipette filler. Show the filled pipette to the supervisor.
2. Practice emptying of the pipette to the last measuring mark. Show the emptied pipette to the supervisor.
3. Practice filling a volumetric flask to the measuring mark. Show the filled flask to the supervisor.

4. Practice using analytical scales with the weighing bottle and coins. Show the masses of the weighed coins to the supervisor.