SUMY STATE UNIVERSITY

LABORATORY WORKS

OF

MEDICAL CHEMISTRY

SUMY 2016

LABORATORY WORK № 1

Safety measures in the chemical laboratory. Laboratory equipment. Balance and weighing.

Characteristic of the subject. When accomplishing practical work the students must be able to use the laboratory equipment, know the rules of behavior in the chemical laboratory. The acquired skills can be used further on when carrying out experiments on the biochemistry and pharmacology chairs.

Purposes:

1. To acquaint with the instruction on safety measures in the chemical laboratory.
2. To acquaint with the laboratory equipment.
3. To acquire skills in work with the electric balance.
4. To define what measuring utensils are the most precise.

Instruction on safety measures in the chemical laboratory.

1. One must enter the laboratory and do any work only with the teacher's permission.
2. Work in the laboratories with chemical agents without white gown is forbidden.
3. Before beginning the work you must read the theoretical material on subject.
4. The students are forbidden to perform the work, which is not connected with the subject of the lesson.
5. When heating the solution in the test-tubes it is restricted to direct the opening of the test-tube at yourself or at your colleagues, take the test-tube by the head, because if the possible emission occurs, there can be accidents.
6. It is forbidden to close the test-tube with your finger when shaking the solution. In the cases when it is necessary to smell the

solution or the fragrant, you should with a slight hand movement direct the air stream towards yourself and smell carefully.

7. It is forbidden to taste the chemicals.
8. When taking the concentrated acids and alkalis with the pipettes it is forbidden to suck in the substance with the mouth. In order to avoid such things the pipettes are provided with the rubber bulbs.
9. All the works with substances that give smoke, fog, harmful or with a specific smell vapor and gases must be carried out under the draught.
10. After usage all the instruments, utensils and chemicals must be put on their places. The utensils must be cleaned after the experiment.

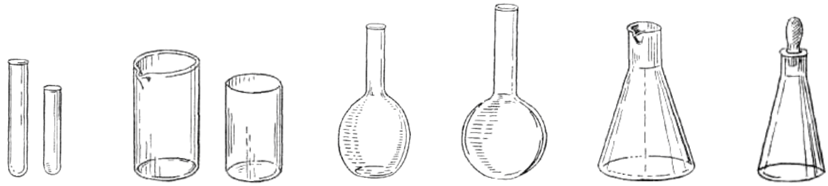
Safety requirements in emergency cases

11. In case of an accident the victim must be given the first help and in case it is needed the doctor must be called for. Before the arrival of the medical worker the victim must be provided with rest and fresh air.
12. In case of thermal burns the burnt place must be sprinkled with sodium bicarbonate (soda), starch, talk or lotion fresh-made of 2% soda or manganese permanganate solutions or neat ethyl alcohol must be applied.
13. In case of burns caused by chemicals, especially by acid (except sulphuric acid) or by alkalis the hurt skin spot must be carefully irrigated with lots of water, then the lotion must be done: when burnt by an acid – 2% soda solution, when burnt by alkalis – 1-2% acetic acid solution.
14. If acid hurts the eyes, they must be washed thoroughly with lots of water and a 3% soda solution.
15. If the hands or other body parts are cut with glass the small glass pieces must be taken away from the body, then the wound must be irrigated by the 2% potassium permanganate solution or ethyl alcohol, smeared with tincture of iodine and bandaged.

Theoretical material

Laboratory equipment and its usage.

Chemical utensils are manufactured from the glass of special types that can bear significant fluctuation in temperature. Two kinds of utensils are distinguished: the thin-walled (Fig.1) and the stout-walled (Fig.2).



1 2 3 4 5 6

Figure 1 The thin-walled utensils.
1 – test-tube, 2 – beaker, 3, 4, 5 – flasks: flat-bottom, round-bottom, conical,
6 – weighing bottle

1 2 3

Figure 2 The stout-walled utensils.
1 – bottle for substances storage, 2 – conical funnel, 3 – separating funnel

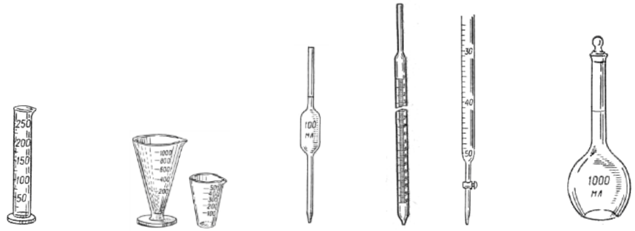
The thin-walled utensils comprise test-tubes, beakers, flasks.

Test-tubes are used when carrying out the experiments with the small quantities of substances.

Beakers are used for preparing and temporary storing the solutions.

Flat-bottom flasks are used for storing distilled water, dissolvents, solutions. They cannot be used for carrying out the experiments connected with strong heating.

Round flasks are used for carrying out the reactions connected with strong heating.
Conical flasks are used for titration of solutions.
Weighing bottles are used for firing the substances in the furnaces.
The stout-walled utensils are used for carrying out the experiments that are not connected with heating.
Measuring glass is used for measuring the volume of a substance.
The measuring glass (Fig.3) comprises measuring cylinders, thistle tubes, pipettes, burettes, graduated flasks.

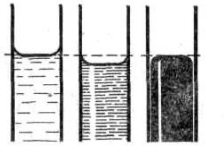


1 2 3 4 5

Figure 3 Measuring glass.
1 – measuring cylinder, 2 – graduated beaker, 3 – pipettes, 4 – burette,
5 – graduated flask.

Measuring cylinders and graduated beaker are used for measuring definite volumes of liquids. In measuring cylinders the density of the solutions can also be defined. There exist 10, 25, 50, 100, 250, 300, 1000 ml cylinders.
Pipettes are used for measuring small volumes of liquids to within 0.005 ml. There exist graduated and ungraduated pipettes. Ungraduated pipettes can be used to measure only such a volume of a liquid that they are designed for. Graduated pipettes can be used to measure any volume. Pipettes are graduated taking into account the outflow of a liquid, so one mustn't blow the rest of the liquid out of them.
Burettes are used for measuring exact volumes of liquids in the quantitative analysis (for titration). With the help of burettes one can

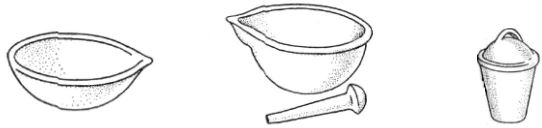
measure volumes of liquids to within 0.3-0.5 ml (for common burettes) and 0.005 ml (for microburettes).
Graduated flasks are flasks with the elongated neck with a mark on it in the shape of a thin ring that defines the volume of a flask. Graduated flasks are used for preparing solutions of exact concentration. There exist 25, 50, 100, 200, 250, 500, 1000 ml flasks.
The level of a liquid in the cylinders, pipettes, burettes can be defined by the mark (Fig.4).



а) б) в)

Figure 4. Position of the meniscus.
a) for a transparent liquid
b) for an opaque or a coloured liquid
c) for a liquid that does not moisten glass.

When filling the utensils with the liquids the eyes of the observer must be the same level with the mark on the flask.
Except the glass utensils porcelain utensils can also be used in the laboratory: basins, mortars, crucibles (Fig.5).



1 2 3

Figure 5 Porcelain utensils.
1 – evaporating basin, 2 – mortar with pestle, 3 – crucible

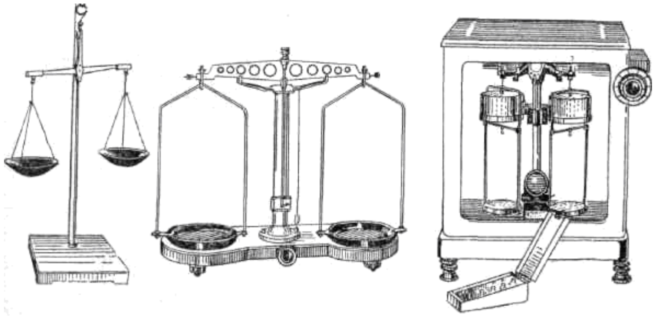
Evaporating basin is used for evaporating liquids and calcinations of substances.
Mortar with pestle is used for grinding solid substances.
Crucible is used for firing substances in the furnaces.
When accomplishing the laboratory works of great importance is optional equipment (Fig.6).

1 2 3

Figure 6 Optional laboratory equipment and instruments.
1 – laboratory support, 2 - test-tube holder, 3 – spatula

Laboratory support with the set of tabs, tings is used for fixing various equipment, flasks, test-tubes, refrigerators.
Test-tube holder is used for fixing test-tubes when they are heated. First of all, the whole test-tube is heated, and then its bottom with a substance.
Spatula is used for laying or removing small quantities of friable solid substances.
Heating instruments are the electric ovens, spirit-lamps. One can use the dry fuel, firing it on the special supports.
Thermometer is an instrument for temperature measurement.
Aerometer is an instrument for liquids density measurement.
In order to weigh substances one uses *balance* – pharmaceutical, apothecary, electric and analytical ones.

Pharmaceutical balance is used for weighing substances within 100 g weight.
Apothecary balance is more precise than the pharmaceutical one. The preciseness of weighing is to within 0.01 g.
The preciseness of weighing with the help of analytical balance is 0.0001 g.



1 2 3

Figure 7 The balance.

Instructions

1. Weigh the weighing bottle and write down the data got into the table.
2. With the help of the measuring cylinder measure 10 ml of water, pour it into the weighing bottle and weigh it. Put down the results into the table and calculate the water mass measured with the cylinder.
3. Pour away the water from the weighing bottle, dry the weighing bottle with the filtered paper.
4. Once again measure 10 ml of water with the help of the measuring cylinder and weigh it. Calculate the average value of 10 ml water, measured with the cylinder.
5. Pour 10 ml of H_2O from the burette into the dry weighing bottle, weigh it. Repeat the experiment twice.
6. Pour 10 ml of H_2O from the pipette into the dry weighing bottle, weigh it. Repeat the experiment twice.
7. After finishing the work put your place in order.

Recording of the experiment

Table 1

Empty weighing bottle mass: $m_{weighing\ bottle}$ =						
No.	Cylinder		Burette		Pipette	
	$m_{weighing\ bottle\ with\ water}$	m_{water}	$m_{weighing\ bottle\ with\ water}$	m_{water}	$m_{weighing\ bottle\ with\ water}$	m_{water}
1 2						
$m_{average}$	–		–		–	
Absolute error	–		–		–	
Relative error	–		–		–	

Calculate the value of the absolute error and relative error, if the theoretic water mass $m_{theoretic}$ = 10.00 g.

$$\text{Absolute error} = |m_{theoretical} - m_{practical}|$$

$$\text{Relative error} = \frac{\text{absolute error}}{m_{theoretical}} \cdot 100\%$$

Conclusions

On the basis of the results got draw a conclusion, what measuring glass was the most precise?

LABORATORY WORK № 2
Coordination compounds

Purposes: 1. To aquaint with the different types of coordination compounds (complex).
2. To aquaint with the chemical properties and preparation of complex compound.

Experiment 1. ***Preparation and properties of complex compound of copper***

(I) Test for copper (II) sulfate.

Take two test-tubes. Place 4-8 drops of copper (II) sulfate $CuSO_4$ solution into each test-tube. Add 2-3 drops of barium chloride $BaCl_2$ into the first test-tube. Observe the formation of white precipitate. In the second test-tube place the piece of tin *Sn*. Tin dissolves gradually and red-brown copper is displaced. Write the reaction equations.

$CuSO_4 + BaCl_2 \rightarrow$ ________________________________

$CuSO_4 + Sn \rightarrow$ ____________________________________

(II) Preparation of complex of tetraamminecopper (II).

Place 10-15 drops of $CuSO_4$ solution in a clean test-tube. Add a few drops of ammonia solution NH_4OH. At first we can observe the formation of blue-green precipitate of $(CuOH)_2SO_4$ which will be dissolved in the excess of NH_4OH with the formation of $[Cu(NH_3)_4]^{2+}$ ions colored blue. Write the reaction equations and balance it.

$CuSO_4 + NH_4OH \rightarrow (CuOH)_2SO_4 + ...$ ________________

$(CuOH)_2SO_4 + NH_4OH \rightarrow [Cu(NH_3)_4]SO_4 +$

$+[Cu(NH_3)_4](OH)_2 + ...$ ___________________________

(III) Properties of complex compound of copper.

Solution of complex of tetraamminecopper (II) divide into two equal parts. Add 2-3 drops of $BaCl_2$ into the first test-tube and *Sn* into the second one. Observe the formation of white precipitate in the first test-tube and absence of free copper in the second one.

$[Cu(NH_3)_4]SO_4 + BaCl_2 \rightarrow$ ____________________________

Experiment 2. ***Complex compound in the exchange reactions***

Take two test-tubes. Place 4-5 drops of $K_4[Fe(CN)_6]$ solution into each test-tube. Add 4-5 drops of $CuSO_4$ solution into the first test-tube. In the second test-tube add 4-5 drops of $FeSO_4$. Observe the formation of precipitate. Write the reaction equations.

$K_4[Fe(CN)_6] + CuSO_4 \rightarrow$ ______________________________

$K_4[Fe(CN)_6] + Fe_2(SO_4)_3 \rightarrow$ ____________________________

Experiment 3. ***Preparation of hydroxocomplex compounds***

(I) Place 8-10 drops of $AlCl_3$ solution in a test-tube. Add a few drops of $NaOH$. Observe the formation of white precipitate of aluminium hydroxide $Al(OH)_3$ which is amphoteric, dissolving in excess $NaOH$. Write the reaction equations.

$AlCl_3 + NaOH \rightarrow$ ____

$Al(OH)_3 + NaOH \rightarrow$ ____________________________________

(II) Repeat analogical experiments with solutions of $Cr_2(SO_4)_3$. Write the reaction equations.

$Cr_2(SO_4)_3 + NaOH \rightarrow$ __________________________________

$Cr(OH)_3 + NaOH \rightarrow$ ____________________________________

LABORATORY WORK № 3

Solutions

Purposes: Preparation of solution.

How would you prepare a 50g of 1% solution $K_2Cr_2O_7$?

1. Calculate the number of grams $K_2Cr_2O_7$ contained in solution.
2. Calculate the volume of water $\left(\rho_{H_2O} = 1\ g/ml\right)$.
3. Put the data into Tab.1

mass of solution	percent concentration	mass of solute m ($K_2Cr_2O_7$)	mass and volume of water m (H_2O), V (H_2O)	density (ρ)	Molar concentration C_M	Molal concentration C_m
50g	10%					

4. Weigh out $K_2Cr_2O_7$ and take distilled water into the measuring cylinder. Dissolve the salt in water into the breaker.
5. Pour the solution from the beaker into the cylinder.
6. Measure the density of solution by aerometer.
7. Calculate the molarity and molality of this solution.

LABORATORY WORK №4
Oxidation – Reduction Reactions

Objective: experimentally investigate redox process.

Experiment 1. ***$(NH_4)_2Cr_2O_7$ Decomposition***

Instructions

Put some microspatulas of $(NH_4)_2Cr_2O_7$ into a test – tube. Heat the bottom of the test – tube for 0.5 minute. Observe the appearance of green flakes of Cr_2O_3 and the evolution of N_2 with water vapor.

How to fix the experimental data

1. Write the equation of the reaction. Write the electron balance and put the coefficients and point out the oxidation and the reductant.

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Experiment 2. ***Medium influence on redox process***

Instructions

1. Take three test – tubes. Put 3–4 drops of $KMnO_4$ solution into each test – tube.
2. Produce a respective medium in each test – tube adding 2–3 drops of H_2SO_4 ($pH < 7$) into the first one; 2–3 drops of distilled water ($pH = 7$) into the second one; 2–3 drops of KOH ($pH > 7$) solution into the third one.
3. Add 2 microspatulas of cristal KNO_2 into each test – tube and stir up to the full solution of the cristals.
4. Observe the change of the coloring in each test – tube. Pay attention to the tube with the alkaline medium as the initial coloring changes very quickly as the result of desproportion.

How to fix the experimental data

1. Write the reactions of reduction of $KMnO_4$ with KNO_2 in the acid, neutral and alkaline media. First, consider that under the experimental conditions KNO_2 is oxidized up to KNO_3; second,

note that manganese compounds have different coloring due to the oxidation state:

- permanganate – anion MnO_4^- in a diluted solution is pink, but with strengthening of may change up to violet (purple);
- manganate – anion MnO_4^{2-} is bright green;
- oxide MnO_2 – insoluble compound of a brown coloring.

Write the equations of electron balance, put the coefficients, point out the oxidant and the reductant, the processes of oxidation and reduction:

In the acid medium:

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In the neutral medium:

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In the alkaline medium:

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Experiment 3. ***$K_2Cr_2O_7$ reduction with ethyl alcohol***

Instructions

Put 5–6 drops of $K_2Cr_2O_7$ solution into a test – tube; add 2–3 drops of concentrated sulfuric acid H_2SO_4 with density 1.84 g/ml. Add 4–5 drops of ethyl alcohol C_2H_5OH and observe the change of the coloring and smell the specific odor.

How to fix the experimental data

1. Write the equation of $K_2Cr_2O_7$ reduction with ethyl alcohol up to acetaldehyde CH_3COH

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