LABORATORY EXERCISE NO. 4
PREPARATION OF HYDROPHOBIC COLLOIDAL SYSTEMS AND DETERMINATION OF A COAGULATION THRESHOLD

Part I

Objective: Preparation of ferric hydroxide sol $Fe(OH)_3$, copper ferrocyanide $Cu_2[Fe(CN)_6]$ and ferric ferrocyanide (Prussian blue sol) $Fe_4[Fe(CN)_6]_3$ by chemical condensation method. Preparation of colophony sol and sulfur sol by physical condensation method. Determination of the sign of the charge of colloidal particles.

Experiment

Chemical condensation

1. Hydrolysis reaction. Ferric hydroxide sol $Fe(OH)_3$. 3 drops of $FeCl_3$ saturated solution are added to 100 mL of boiling distilled water. The hydrolysis of trivalent iron ions will occur and molecules of ferric hydroxide are produced and colloidal particles are condensed. The color of $Fe(OH)_3$ sol is dark brown. The sign of the charge of colloidal particles is determined by method of capillary analysis.

2. Ion exchange reactions.

a) Copper ferrocyanide sol $Cu_2[Fe(CN)_6]$. Approximately 10–12 drops of 20 % potassium ferrocyanide $K_4[Fe(CN)_6]$ solution are diluted up to 100 mL by distilled water and 3-5 drops of copper sulfate $CuSO_4$ solution are added at shaking. Brown $Cu_2[Fe(CN)_6]$ sol is produced.

b) Ferric ferrocyanide (Prussian blue sol) $Fe_4[Fe(CN)_6]_3$. 10–12 drops of 20 % potassium ferrocyanide $K_4[Fe(CN)_6]$ solution are diluted by water up to 100 mL. Drop of saturated ferric chloride $FeCl_3$ solution is added to diluted $K_4[Fe(CN)_6]$ solution at shaking. Dark blue transparent Prussian blue sol is produced. The sign of the charge of colloidal particles is determined by method of capillary analysis.

Physical condensation (solvent replacement)

1. Colophony sol. 2–4 drops of colophony solution in ethyl alcohol are added to 50 mL of distilled water at shaking. Colophony hydrosol with negatively charged colloidal particles is produced.

2. Sulfur sol. 4–5 mL of saturated sulfur solution in ethyl alcohol is added to 50 mL of distilled water at shaking. Bluish (in passing light yellowish) sulfur sol with negatively charged colloidal particles is produced.

Tyndall effect may be observed for obtained hydrosols using special device.

Determination of the sign of the charge of colloidal particles (granules) by method of capillary analysis

The sign of the charge of colloidal particles (granules) of tinted sols can be determined by method of capillary analysis. Capillaries surface of filter paper is used as charged surface. The surface of the cellulose capillaries are negatively charged (because of dissociation of cellulose hydroxyl groups) and positively charged hydrogen ions are collected in water close-fitting to them. Water moistens cellulose very good and a concave meniscus forms in paper capillaries. «An effect of capillary rise» arises as result of action of surface-tension forces: water rises on filter paper capillaries.

If negatively charged colloidal particles are presented in water, they will push off from the same sign charged paper surface and they will rise together with water. If colloidal particles are positively charged, they will attract by oppositely charged paper surface and will remain on it.

Ferric hydroxide sol $Fe(OH)_3$ and Prussian blue sol $Fe_4[Fe(CN)_6]_3$ are poured into small vessels and filter papers are immersed in these vessels. The rising height of water and sols is compared after 3–5 minutes.

Write in your copybook the following:

1. The reactions of sols preparation by chemical condensation. Write the micelles structure of these sols. Show aggregate, nucleus, granule, potential-determining ions, Stern layer and diffuse layer of counter-ions.

2. Make conclusion about the charge of colloidal particles of ferric hydroxide and ferric ferrocyanide by using data of capillary analysis.

Part II

Objective: Determination of the coagulation thresholds of $Fe(OH)_3$ hydrosol by two electrolytes with various charges of anions.

Experiment and calculations

Determination of coagulation thresholds of $Fe(OH)_3$ *hydrosol by electrolytes.* Take 11 test-tubes. Prepare solutions according to Table 1 data. Distilled water and electrolyte solution are added in different quantities from burettes into each test-tube. Volume should be 5 mL in sum (for example, 3 mL of water + 2 mL of electrolyte solution). Potassium sulfate solution K_2SO_4 (0.003 mol/L) is used for one row of test-tubes. Potassium ferricyanide solution $K_3[Fe(CN)_6]$ (0.0005 mol/L) is used for other row of test-tubes. 5 mL of $Fe(OH)_3$ sol is quickly added into all test-tubes. Test-tubes content are mixed by shaking and are left on 15 minutes. 5 mL of distilled water and 5 mL of $Fe(OH)_3$ sol are mixed that to prepare *the control solution*. Over 15 minutes the solutions are compared with content of the control solution. Coagulation characteristic is turbidity of solution in comparison with control solution. Results are denoted as «+», if the coagulation is observed; and as «–», if the coagulation is not observed.

Table 1. Solutions volumes, mL

Solutions	Test-tube number and volumes, mL				
	1	2	3	4	5
Distilled water	4.7	4.5	4.0	3.5	3.0
K_2SO_4, 0.003 mol/L	0.3	0.5	1.0	1.5	2.0
$Fe(OH)_3$ sol	5.0	5.0	5.0	5.0	5.0
Result: «+» *or* «–»					
Distilled water	4.7	4.5	4.0	3.5	3.0
$K_3[Fe(CN)_6]$, 0.0005 mol/L	0.3	0.5	1.0	1.5	2.0
$Fe(OH)_3$ sol	5.0	5.0	5.0	5.0	5.0
Result: «+» *or* «–»					

Calculation of coagulation thresholds

For each electrolyte, i.e. K_2SO_4 and $K_3[Fe(CN)_6]$, calculate electrolyte moles number for the test-tube where coagulation is observed and that contain minimal electrolyte solution volume

$$n = c_{electrolyte} V_{\min}\text{, mmol,} \tag{1}$$

where $V_{\min}$ – volume of electrolyte solution in this test-tube in mL.

Calculate the coagulation thresholds by the equation

$$\gamma = n/V\text{, mmol/L,} \tag{2}$$

where V – total volume of solution in the test-tube, in present experiment it equals 0.01 liter.

Make conclusions about agreement of results obtained and Schultze–Hardy rule taking into account that the salts used at dissociation produce univalent potassium ions K^+ and divalent SO_4^{2-} or trivalent $[Fe(CN)_6]^{3-}$ anions.