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Supporting information for article:

From geology to biology: an interdisciplinary course in crystal growth

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Appendix 1. Selected links to the sites of museums and exhibitions related to crystals (all sites were accessible on the date of submission of the last revision, 29/07/22, at least from Russia, accessibility of some sites may depend on the IP-address of a device)

1. NSU Research and Education Center “Evolution of the Earth” (Novosibirsk, Russia)
https://education.nsu.ru/earth_evolution/en/
2. Central Siberian Geological Museum (Novosibirsk, Russia); Центральный Сибирский геологический музей (Новосибирск) <https://www.igm.nsc.ru/index.php/ob-institute/struktura/museum>
3. A. E. Fersman Mineralogical Museum of Russian Academy of Sciences (Moscow, Russia); Минералогический музей имени А. Е. Ферсмана РАН (Москва)
https://fmm.ru/Main_Page?setlang=en
4. Mineralogical Museum of St-Petersburg State University (St-Petersburg, Russia); Минералогический музей Санкт-Петербургского государственного университета
<http://mmus.geology.spbu.ru/>
5. Mining museum (St-Petersburg, Russia) <https://museum.spmi.ru/en>
6. Mineralogical Museum “Planet” (Ekaterinburg, Russia); Минералогический музей «Планета» (Екатеринбург) <https://museum-planeta.ru/>
7. Ural Geological Museum of the Ural State Mountain University (Ekaterinburg, Russia); Уральский геологический музей - ФГБОУ ВО "Уральский государственный горный университет" <http://ugm.ursmu.ru/>
8. Natural Sciences Museum of Ilmen National Park (Miass, Russia); Естественно-научный музей Ильменского заповедника, Миасс
https://museum.chelscience.ru/?page_id=3168
9. Museum of Stones (Ekaterinburg, Russia); Екатеринбургский музей камня
<https://vsemuzei.com/rossiya/muzej-kamnya-i-mineralov-v-ekaterinburge>
10. A. V. Sidorov State Mineralogical Museum of the Irkutsk State Research Technical University (Irkutsk, Russia); Государственный Минералогический Музей имени А.В. Сидорова («Иркутский национальный исследовательский технический университет»)
<http://mineral.inrtu.ru/>
11. I. V. Belkov Museum of Geology and Mineralogy of the Russian Academy of Sciences (Apatity, Russia); Музей геологии и минералогии им. И.В. Белькова ГИ КНЦ РАН (Апатиты) <http://museum.ru/M2304>

12. Geological Museum (Novokuzneck, Russia); Геологический музей (Новокузнецк)
<https://russia.travel/objects/314337/>; <http://geofondkem.ru/sources/museum-60.pdf?ysclid=l66bmh313p650039147>
13. I. K. Vazhenov Mineralogical Museum (Tomsk, Russia); Минералогический музей имени И.К. Баженова (Томск) <https://www.tsu.ru/university/museums/minmuseum.php>
14. Mineralogical Museum of Tomsk Technical University (Tomsk, Russia); Минералогический музей (Томский политехнический университет)
<https://tpu.ru/university/meet-tpu/excursion>
15. Kolyvan Museum of the History of Stone-Cutting in Altai (Kolyvan, Russia); Колыванский музей истории камнерезного дела на Алтае <http://museum.ru/M1497>
16. Geological Museum of the Republican National Geological Center (Minsk, Belarus); Геологический музей Республиканского унитарного предприятия «Научно-производственный центр по геологии» (Беларусь)
<https://geologiya.by/%D0%BC%D1%83%D0%B7%D0%B5%D0%B9/?ysclid=l6697w8v8g801644116>
17. Mineralogisches Museum - Philipps-Universität Marburg <https://www.uni-marburg.de/en/fb19/minmus>
18. Mineralogical Collection Germany at the Krügerhaus (Freiberg) <https://www.terra-mineralia.de/english/collections/mineralogical-collection-germany>
19. Museum Mineralogia München und Mineralogische Staatssammlung
<https://www.mineralogische-staatssammlung.de/index.php/en/>
20. Museum für Naturkunde Leibniz-Institut für Evolutions- und Biodiversitätsforschung
<https://www.museumfuernaturkunde.berlin/en>
21. Museum für Mineralogie und Geologie (Senckenberg Gesellschaft für Naturforschung) <https://www.senckenberg.de/de/institute/senckenberg-naturhistorische-sammlungen-dresden/museum-fuer-mineralogie-und-geologie/>
22. Oberharzer Bergwerksmuseum <https://www.oberharzerbergwerksmuseum.de/en/>
23. Kristalle: Geologisch-Mineralogische Ausstellung der ETH-Zürich
<http://www.kristalle.ch/sammlung/slidesethz.asp>
24. Naturhistorisches Museum Wien https://www.nhm-wien.ac.at/minerale_oesterreichs
25. Cockburn Geological Museum (The University of Edinburgh)
<https://www.ed.ac.uk/visit/museums-galleries/geology>
26. National Museum of Scotland <https://www.nms.ac.uk/national-museum-of-scotland/>

27. Mineralogy collections (Natural History Museum, London)
<https://www.nhm.ac.uk/our-science/collections/mineralogy-collections.html>
28. Mineral sciences collection (South Australia Museum)
<https://www.samuseum.sa.gov.au/collection/mineral-sciences>
29. Australian Minerals <https://www.bukartilla.com.au/australian-minerals-summary>
30. Rocks Discovery Museum (Sydney) <https://rocksdiscoverymuseum.com/>
31. Museo Geominero- Instituto Geológico y Minero de España
<http://www.igme.es/museo/>
32. Musée de Minéralogie des Mines <https://www.musee.minesparis.psl.eu/Home/>
33. Musée de Bourg d'Oisans <http://www.musee-bourgdosans.fr/>
34. Mineral Hall (Royal Belgian Institute of Natural Sciences)
<https://www.naturalsciences.be/en/museum/exhibitions-view/240/398/395>
35. Museu Geológico de Lisboa <https://www.mindat.org/museum-338.html>
36. Oxford University Museum of Natural History <https://oumnh.ox.ac.uk/mineralogy-and-petrology>
37. Design museum presents digital crystal exhibition:
<https://www.montcalm.co.uk/blog/design-museum-presents-digital-crystal-exhibition/>
38. “Crystal Dome”:
https://kristallwelten.swarovski.com/Content.Node/wattens/Swarovski_Exhibition.en.html

Appendix 2. Selected references to the publications that can be used when preparing the topics listed in Tables 1-3

Basics crystal growth

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Appendix 3. Educational courses given at the Chair of Solid State Chemistry of Novosibirsk State University that can be taken by students complementary to the course on Crystal Growth

General courses:

- Solid State Chemistry – for all 3rd year chemistry students, 51 hours* lectures, 51 hours practicals, includes crystallography and crystal chemistry
- Supramolecular Chemistry - for all 4th year chemistry students, 20 hours lectures

Courses of choice:

- Introduction into diffraction techniques. Basics - 17 hours lectures, 17 hours practicals
- Introduction into diffraction techniques. Advanced level - 17 hours lectures, 17 hours practicals
- Introduction into structural biology - 16 hours lectures, 16 hours practicals
- Materials for large-scale facilities - 16 hours lectures, 16 hours practicals
- Low-dimensional materials - 16 hours lectures, 16 hours practicals
- Introduction into the structural databases -12 hours lectures, 22 hours practicals
- Introduction into synchrotron and neutron techniques -32 hours lectures
- From drug substances to drug formulations - 18 hours lectures, 18 hours practicals
- Materials for energy and catalysis 16 hours lectures, 16 hours practicals
- Physico-chemical analysis in solid state chemistry and materials sciences -20 hours lectures, 12 hours laboratory training
- Introduction into materials sciences 16 hours lectures, 16 hours practicals
- High-pressure techniques 16 hours lectures, 16 hours practicals

* - throughout the text, we refer to course hours. Homework is not taken into account

Appendix 4. Selected links to the resources related to teaching crystal growth at different levels and for different audiences available on-line

Practical aspects of crystal growth for students and general public:

1. Project “Fascinating Science” for children; Проект “Занимательная наука для школьников” <https://cfc.nsu.ru/?author=3>
2. Crystals; <http://zircon81.narod.ru/Metodica.html>
3. How to grow a single crystal: https://www.youtube.com/watch?v=cNyQ_pMGxWs
4. A project "Silicate garden" <https://infourok.ru/proekt-po-himii-silikatniy-sad-1241109.html>
5. Tips and tricks (techniques of crystallization):
http://www.chemistryviews.org/details/education/2532131/Tips_and_Tricks_for_the_Lab_Growing_Crystals_Part_1.html
; http://www.chemistryviews.org/details/education/2538901/Tips_and_Tricks_for_the_Lab_Growing_Crystals_Part_2.html ;
http://www.chemistryviews.org/details/education/2538941/Tips_and_Tricks_for_the_Lab_Growing_Crystals_Part_3.html ;
http://www.chemistryviews.org/details/education/4519261/Tips_and_Tricks_for_the_Lab_Air-Sensitive_Techniques_4.html

Crystal growth & crystallography

1. Crystal gallery IUCr: <https://www.iycr2014.org/participate/crystal-growing/gallery> & <https://www.iucr.org/education/resources>
2. “The Fascination of Crystals and Symmetry” project: <https://crystalsymmetry.wordpress.com/230-2/>
3. Training and Educational Resources CCDC: <https://www.ccdc.cam.ac.uk/Community/educationalresources/>

Useful video:

General:

1. “Beautiful Chemistry” project: <https://www.beautifulchemistry.net/reaction>
2. Crystal Growth: https://www.youtube.com/watch?v=Nt5TA2_ABJg

Crystallization of inorganic compounds:

1. KDP crystal growth: https://www.youtube.com/watch?v=l_USYub3djY
2. Timelapse shows frost through a microscope: <https://www.youtube.com/watch?v=bDPczGUovzE>
3. Microscopic time-lapse of growing snowflake: <https://www.youtube.com/watch?v=MCA2VmDVzEo>
4. The beauty of crystal growth (crystallization of sodium acetate): <https://www.youtube.com/watch?v=hibmI6aGOso>
5. Hot ice (sodium acetate) beautiful science experiment: <https://www.youtube.com/watch?v=BLq5NibwV5g>
6. Experiment - salt crystals growing - time lapse: <https://www.youtube.com/watch?v=0g7qYPZHf0A>
7. Time lapse video of epsom salt crystals growing under polarized light: <https://www.youtube.com/watch?v=6feLRBBtC9c>
8. Envisioning Chemistry Crystallization2: <https://www.youtube.com/watch?v=dyVz6h2wrEA>
9. Beauty of crystallization. <https://www.youtube.com/watch?v=lo0cp2uhxb0&t=43s>

Crystallization of organic compounds

1. Crystallisation of Vitamin C: <https://www.youtube.com/watch?v=DeJw03BLTGo>
2. Erythritol Crystallization: <https://www.youtube.com/watch?v=oFxz7tQY-Z8>
3. Thymol crystallization: <https://www.youtube.com/watch?v=lfFePrI-UCU>

Biological & macromolecule crystallization

1. Protein crystallography: <https://www.youtube.com/watch?v=8SN1kmfhWcs>
2. Understanding crystallography: part 1 from protein to crystals: <https://www.youtube.com/watch?v=gLsC4wlrR2A>
3. Understanding crystallography: part 2 from crystals to diamond: <https://www.youtube.com/watch?v=WJKvDUo3KRk>
4. Single crystal diffraction familiarization video: <https://www.youtube.com/watch?v=suVNYD1nCm4>
5. Faster and smaller macromolecular crystallography at DLS: <https://www.youtube.com/watch?v=S3g5kXJ8ib8>

6. Seeding mechanism during crystallization:

<https://www.youtube.com/watch?v=asLtlLKPT0k>

7. David J. Haas, part 1: Cryo-cooling Protein Crystals: The First 52 Years:

<https://www.youtube.com/watch?v=Q6Ai4dGml9M>

Metals

1. Gallium - a terminator metal <https://www.youtube.com/watch?v=iPlhdzMKp6A>

2. Beautiful Chemical Reactions - Metal Displacement

<https://www.youtube.com/watch?v=C7ZZWXI6gLs>

3. Envisioning chemistry- metal displacement II

<https://www.youtube.com/watch?v=IT2v7JgiGSY>

4. Envisioning Chemistry- Disappearing Metals

<https://www.youtube.com/watch?v=AcYaQyfgg8o>

5. Microscopic time-lapse- see the crazy chemistry of reacting metal - short film showcase <https://www.youtube.com/watch?v=RSrLwG25euc>

Liquid crystals

1. What are Liquid Crystals? <https://www.youtube.com/watch?v=MUWDwVHVLio>

2. Crystallization of cholesteryl pelargonate

<https://www.youtube.com/watch?v=i8Krw0xOagg>

3. Birefringence and phase transition of the liquid crystal Cholesteryl Pelargonate 97%

<https://www.youtube.com/watch?v=9Ku0MHWt6bw>

Semiconductors

1. Process of obtaining single-crystals of Si: https://youtu.be/8QKzS_w_Ko0

2. Some more about the Czochralski method: <https://youtu.be/xftnhfa-Dmo>

3. p- and n-conductivity and transistors: <https://youtu.be/jh2z-g7GJxE>

4. Wafer manufacturing process: <https://youtu.be/3TOpg1niATg>

5. Silicon vs Germanium. Differences between Si and Ge:

<https://www.youtube.com/watch?v=iR62tf4NkcA>

“Chemical garden”:

1. Chemical garden https://en.wikipedia.org/wiki/Chemical_garden

2. «Chemical garden» https://elementy.ru/kartinka_dnya/224/Khimicheskiy_sad

3. The Chemical Garden - A time lapse movie

<https://www.youtube.com/watch?v=z4SiKBfFRbE>

4. Envisioning Chemistry- Chemical Garden

II: <https://www.youtube.com/watch?v=JslO9tiBaSw>

Miscellaneous:

1. Spherulite Formation: <https://www.youtube.com/watch?v=9qbCyJ-nF5A>

2. Envisioning Chemistry- Getting Hot (with Thermal Imaging):

<https://www.youtube.com/watch?v=VSyEWg10WjM>

3. Envisioning Chemistry- Precipitation IV:

<https://www.youtube.com/watch?v=603k16Rx7YU>

4. Crystals under microscope 2: <https://www.youtube.com/watch?v=SsvJtKNrsj8>

5. Timelapse of Crystals Growing: <https://www.youtube.com/watch?v=eHfO8pmaXMg>

YouTube channels:

1. Beauty of Science:

<https://www.youtube.com/channel/UCYmL90kuJusvrpVv3B60Rsg>

2. International Union of Crystallography <https://www.youtube.com/user/theIUCr>

3. BioXFEL <https://www.youtube.com/user/BioXFEL>

4. Rigaku Corporation <https://www.youtube.com/c/RigakuCorporation>

5. CrystalGrower https://www.youtube.com/channel/UCnftdepWN2iAt_hz8s2WhQg

6. Frank Hoffmann <https://www.youtube.com/c/FrankHoffmann1000>

7. Distant learnig at the Faculty of Materials Sciences of the Moscow State University;
ΦHM MFY <https://www.youtube.com/channel/UCepiCWoe2ew8ID901LL4Fkg>

Simulators and games:

1. vDiffraction: A serious game about diffraction and crystals

<https://www.ill.eu/users/support-labs-infrastructure/software-scientific-tools/vdiffraction/>

2. EwaldSphere (simulates a 3-circle single-crystal X-ray diffractometer)

<http://academic.sun.ac.za/barbour/Software.html>

Virtual tours to large-scale facilities:

1. ILL <https://www.ill.eu/about-the-ill/documentation/films-animations/ill-virtual-tour>

2. BESSY II https://www.helmholtz-berlin.de/zentrum/aktuell/mediathek/360/panoramen_en.html#c504498 ;
https://www.helmholtz-berlin.de/zentrum/aktuell/mediathek/360/index_en.html
3. «Nauka 360°»: <https://360.rscf.ru/#projects>
4. SuperrAIntgenlaser XFEL - Lebensbausteine im Blick - Projekt Zukunft
<https://www.youtube.com/watch?v=oLTL3sJqK4U>
5. Der neue RAIntgenlaser European XFEL - Tunnelflug
<https://www.youtube.com/watch?v=p3G90p4glQA>
6. FLASH - DESY's High-Speed Camera for the Nanocosmos
<https://www.youtube.com/watch?v=hC6TT-0gQcU>

Appendix 5. Sample plans of selected lectures

5a. General introduction. Crystals from geology to biology

Different types of crystals. Relation between crystal structure and the type of chemical bonds and interactions. Examples of very large and very small crystals.

Different definitions of a crystal. How and why they changed with time. 1D, 2D, 3D crystals.

Parameters to be controlled on crystal growth:

Chemical formula

Polymorph

Size

Shape

Perfection / defects (types, concentration, spatial distribution)

When we need to prevent crystallization.

Stable, metastable, unstable forms.

Thermodynamics vs kinetics.

Classical / non-classical nucleation and growth.

Crystallization techniques (an overview):

From solution

From melt

From gas

As a result of a chemical reaction (also a solid-state reaction)

Electrocrystallization

Biocrystallization

Effects of electromagnetic fields, or light

High-pressure crystallization

Confined crystallization

Crystallization in levitating drops

Crystals in physics, chemistry, planetary sciences, biology, medicine.

Crystals used as materials and materials helping to study crystals.

Crystals and food. Chocolate, Ice-cream, Honey.

5b. Defects in crystals and crystal growth

Part 1. Classification of defects in crystals.

Zero-dimensional (point defects), one-dimensional (dislocations), two-dimensional, and three-dimensional.

Equilibrium concentration of point defects, temperature dependence. Typical values of concentration of point defects.

Edge and screw dislocations.

Part 2. Sources of dislocations in grown crystal.

Growth of dislocations from a seed crystal, uneven distribution of point defects, thermal and mechanical stresses, "mistakes" of growth (for example, the meeting of two growth islands with different orientations).

Typical values of concentration of dislocations in different crystalline materials (carefully grown high purity single crystal, annealed single crystal, annealed polycrystal, cold worked metal).

Part 3. The importance of point defects for the creation of functional **single-crystalline** materials.

Semiconductors with p- and n-type of conductivity.

Photo- and thermoluminescent materials with impurity luminescence.

5c. Melt crystallization and solid-state growth in Nature

Part 1. Crystal growth from silicate melt in the Earth's crust

Minerals and rocks; rock texture, inter-grain boundaries, idiomorphic and xenomorphic relations of minerals.

Deep (intrusive) and superficial (effusive) magmatic rocks. Granite and basalt as representative constituents of the Earth's crust, their mean chemical and mineral composition.

P-T conditions of the generation and crystallization of silicate magmas in the Earth's crust. "Wet" and "dry" solidus of the rock: how it determines the migration of magma towards the surface.

Phase diagrams 'diopside – anorthite' and 'k-feldspar – SiO₂' as models of crystallization of silicate melt. Liquidus phase. Restite (eutectic) melt and its textural signs. The transition from idiomorphic to xenomorphic crystals during the crystallization.

The conditions of crystallization in deep magmatic chamber and erupted lava. Cooling rates, temperature gradients, oversaturation. The variation of the rate of crystal nucleation and growth depending on oversaturation. Multistage crystal growth in basaltic melts during its ascent and effusion. The resulting textures of intrusive (granite) and effusive (basalt) rocks.

Co-crystallization from eutectic melt: oriented (graphic) intergrowths of minerals -

pegmatites. The necessary conditions (over-cooling, diffusion and crystal growth rates, the presence of water) for their growth.

Part 2. Solid state growth in the Earth's crust

Exsolution.

Solid solution. Isomorphic elements. Ordering of solid solution, its crystal chemical prerequisites. Exsolution and its thermodynamic interpretation. Solvus, immiscibility range. Exsolution processes in minerals; cooling rates and the degree of separation of the product phases. Homogeneous and heterogeneous nucleation. Energetic parameters of the nucleus surface: elastic strain and surface energy. Equilibrium (chemical) and coherent solvus. Exsolution structures in feldspars.

Metamorphic reactions.

Metamorphic processes in the Earth's crust, pressure and temperature as their principal factors. Lithostatic and tectonic pressure.

Metamorphic reaction or replacement reaction; its isochemical character. Pseudomorphs. Polymorphic transition. Polymorphism in Al_2SiO_5 system.

Impact metamorphism, its P-T parameters. Popigai astrobleme. Impact diamonds – pseudomorphs after graphite. Lonsdeilite (hexagonal diamond) as a sign of impact origin of diamond. Fine structure of lonsdeilite-diamond intergrowths.

5d. High-pressure crystallization

Part 1. Crystallization with diamonds

1.1. Why crystallize at high pressures?

- 1.1.1. New target products (diamonds, other artificial minerals, materials, drugs, chemicals)
- 1.1.2. Understanding structures and processes (chemical engineering, chemical bonds and interactions, theory of crystal growth, structure of solutions and melts, formation and transformations of minerals in the Earth and other planets, biology-related processes)

1.2. How to crystallize at high pressures?

- 1.2.1. Pressure cells (diamond anvil cells, large volume presses, capillaries)
- 1.2.2. Pressure calibration
- 1.2.3. Characterization of samples *in situ* (diffraction, spectroscopy, optical microscopy)
- 1.2.4. Crystallization starting from gases, liquids, solids

1.3. Crystallization starting from gases (oxygen, carbon dioxide, H_2S , gas hydrates)

1.4. Crystallization starting from liquids

- 1.4.1. Pure liquids
- 1.4.2. Solutions of solids in liquids

- 1.4.3. Obtaining phases metastable at ambient pressures and their recovery
- 1.4.4. Thermodynamics
- 1.4.5. Kinetics
- 1.5. Crystallization starting from solids
 - 1.5.1. Solids immersed in a fluid (recrystallization)
 - 1.5.2. Solid mixtures (crystallization on synthesis)
 - 1.5.3. Thermodynamics
 - 1.5.4. Kinetics

Part 2. Crystallization of diamonds

- 2.1. Crystallization of diamonds as target materials
 - 2.1.1. Nanodiamonds
 - 2.1.2. Large single crystals
 - 2.1.3. Doped diamonds for special applications
- 2.2. Crystallization of diamonds, in order to understand the natural processes of their growth. Differences with procedures used in materials sciences.

5e. Biocrystallization

Biopolymers, DNA, RNA, proteins, peptides, nucleotides.

Peptide bond.

Methods of obtaining biopolymers.

Molecular mass and the number of the amino acid residues. Examples of an insulin monomer, lysozyme, influenza virus glycoprotein, eukaryotic small ribosomal subunit (40S). The sizes of hemoglobin and erythrocyte size.

Requirements to a solution of a biopolymer suitable for crystallization.

A general overview of the methods of structure determination of biopolymers with analysis of the Protein Database (PDB) statistics on the number of structures defined by each method.

Alpha Fold2.

The main stages of the crystallization of the biopolymers.

Concepts: the region of the undersaturation, solubility curve, metastable zone, nucleation zone, precipitation zone, the supersaturation region, phase diagrams for the systems where supersaturation is achieved by using an antisolvent.

Methods of the crystallization of biopolymers and different routes of reaching nucleation: Vapor-diffusion method (hanging drop, sitting drop, sandwich drop), batch experiments, dialysis, Free interface diffusion method. The method of counter-diffusion.

«Streak seeding».

Crystallization of membrane proteins.

Equipment for preparing protein solutions, micro- and nano- droplets, crystal growth at a constant desirable temperature.

Chip crystallization.

Appendix 6. Descriptions of selected laboratory exercises

6a. Crystallization of alums

Main goal – crystal growth of potassium alum crystals

Approximate time limit – 2 academic hours plus 4-6 weeks for crystal growth

Tasks

1. Learning how to grow seed crystals
2. Preparation of saturated solution of potassium alums
3. Crystal growth of potassium alums from saturated solution by slow evaporation using seed crystals
4. Description of crystal habit and comparison with the crystals of other substances
5. Preparation of a spherical crystal and observation of faceting in isothermal conditions

Substances and materials

1. Alums
2. Seed crystals of alums
3. Distillated water

Equipment

1. Laboratory coat, goggles, gloves, heating plate, glass beakers (100 ml), glass funnels, filter paper, Petri dish, spatulas, toothpick, thread, sandpaper, microscope.

Description of lab work

Preparation of seed crystals (carried out two days before the class, can be done by assistant)

A solution of potassium alum in distilled water is prepared in the ratio of ~ 5 g per 50 ml with slight heating (~ 50 °C). If needed, some extra amount of the substance is added until an insoluble precipitate is formed. Part of the solution is filtered into a Petri dish and is left to crystallize the seeds (this process may take from several hours to 1 day). It is preferable to use a Petri dish as a crystallization container to increase the evaporation area and hence crystallization rate. In addition, due to the low side wall of the dish, the extraction of crystals from the mother liquor is easier than from a beaker. Once crystallized, the seed crystals are removed from the solution and dried on filter paper.

1. Preparation of saturated solution of potassium alums

Alums are dissolved in 1 L of distilled water until the insoluble precipitate is formed. The solution is then stirred for 15 min and then filtered into a clean glass vessel. This solution will be used immediately, to start crystallization. This same solution will be also needed over the next 4-6 weeks to top-up the mother liquor in the crystallization vessels (see below). The

following concepts are discussed in relation to this laboratory exercise: seed, saturated solution, faceting, habit.

2. Crystal growth

To obtain the best defect-free octahedral crystals, it is necessary to choose the most transparent and well-faceted seed crystals, without internal defects or inclusions (this can be achieved using an optical microscope). The selected crystal is fixed on a thread tied to a stick (toothpick, pencil, etc.) and placed into a saturated solution of potassium alum (Fig. S1a). The container for crystallization is covered with filter paper and left to grow a larger crystal. It is necessary to periodically (once every 3-4 days) check the growth of the crystal to avoid pollution of its surface with small crystals. The appearance of additional seed crystals on the thread and at the bottom of the crystallization container must also be avoided. If necessary, the thread (or the crystal surface) should be cleaned mechanically or using a hot tap water jet (without harming the target crystal). It is also necessary to check the level of the mother liquor in the container and, if it has decreased due to evaporation, top-up the beaker from the previously prepared saturated solution of potassium alum (from Step 1). After extraction from the solution and drying (Fig. 2a, Fig. S1b,c), the crystals must be stored in a sealed container to prevent dehydration, since alum crystallizes as a crystal hydrate.

3. Cutting of a spherical crystal and its growth

A large crystal grown at Step 2 is evenly sanded with sandpaper until it becomes spherical. The spherical crystal is suspended in a saturated solution of potassium alum under isothermal conditions to observe the growth figures of the faces on the surface of the originally spherical sample (2-4 weeks).

Finishing the lab work: wash all the glassware, discuss the obtained results.

6b. Crystallization of L-ascorbic acid (vitamin C) - L-serine co-crystals by slow evaporation

Main goal – crystal growth of co-crystals of L-ascorbic acid with L-serine.

Approximate time limit: 2 academic hours

Tasks:

1. Learning how to grow multicomponent single crystals by slow evaporation from droplets
2. Observing stages of nucleation and crystal growth
3. Practicing the manipulations with seed crystals
4. Preserving and storing co-crystals for further unit cell determination by single crystal X-ray diffraction

Substances and materials:

1. L-serine (105 mg)
2. L-ascorbic acid (176 mg)
3. Distilled water

Equipment:

Laboratory coat, goggles, gloves, plastic test tubes, slide glasses, Parafilm® M, syringes (1 ml) with needles, cotton wool or filter nozzles for a syringe with a pore diameter of 0.45 microns or 0.22 microns, pipette (0.1-1 ml), crystal manipulation kit, cryo-oil, marker.

Description of lab work:

Dissolve 105 mg of L-serine and 176 mg of L-ascorbic acid in 600 ml of water in plastic test tubes. Dissolution takes 10-15 minutes. Cover a glass microscope slide with Parafilm® M, so that part of the glass remains uncovered. The aqueous solution is filtered with a syringe through cotton wool or filter nozzle into a new plastic test tube. A droplet of 50 µl is placed on the glass surface and 3 bigger droplets of ca. 100 µl, 150 µl and 300 µl are put on the surface covered by Parafilm® M. Attention should be paid to the droplet shape. When placed on the glass surface the drop tends to lose its shape. Instead, when a drop is placed on the hydrophobic Parafilm® M surface, the drops maintain an arched shape. The small drop of 50 µl on the glass is used to obtain crystal nuclei that will be taken to grow bigger crystals in drops places on Parafilm® M (see Fig. S2). The following processes and concepts are discussed: solubility curve for a two-component system, change in the concentration of a dissolved compound during solvent evaporation, "metastable zone", nucleation, supersaturation, spontaneous crystallization, co-crystal.

After approximately 40 min from the beginning of the experiment, crystal nuclei from the droplet on the glass surface are transferred using a metal needle into all 3 drops of different volumes (100, 150, 300 µl) located on the Parafilm® M (see Fig. S2). Crystals should begin to grow in the 100 µl droplet, and dissolve in the larger droplets if the humidity is high. Observe the growth of crystals in the 100 µl drop directly through a microscope. After a while, put the nuclei crystals again into drops of 150 µl and 300 µl again. Observe crystal growth or dissolution of the nuclei crystals again. As soon as it is possible to grow crystals larger than 0.2 mm, the crystals are extracted from the drop. Select and preserve the single crystals in cryo-oil for further testing on a single crystal X-ray diffractometer.

Finishing the lab work: wash all the glassware, turn off all equipment, discuss the experience gained, main points of theory and the obtained results.

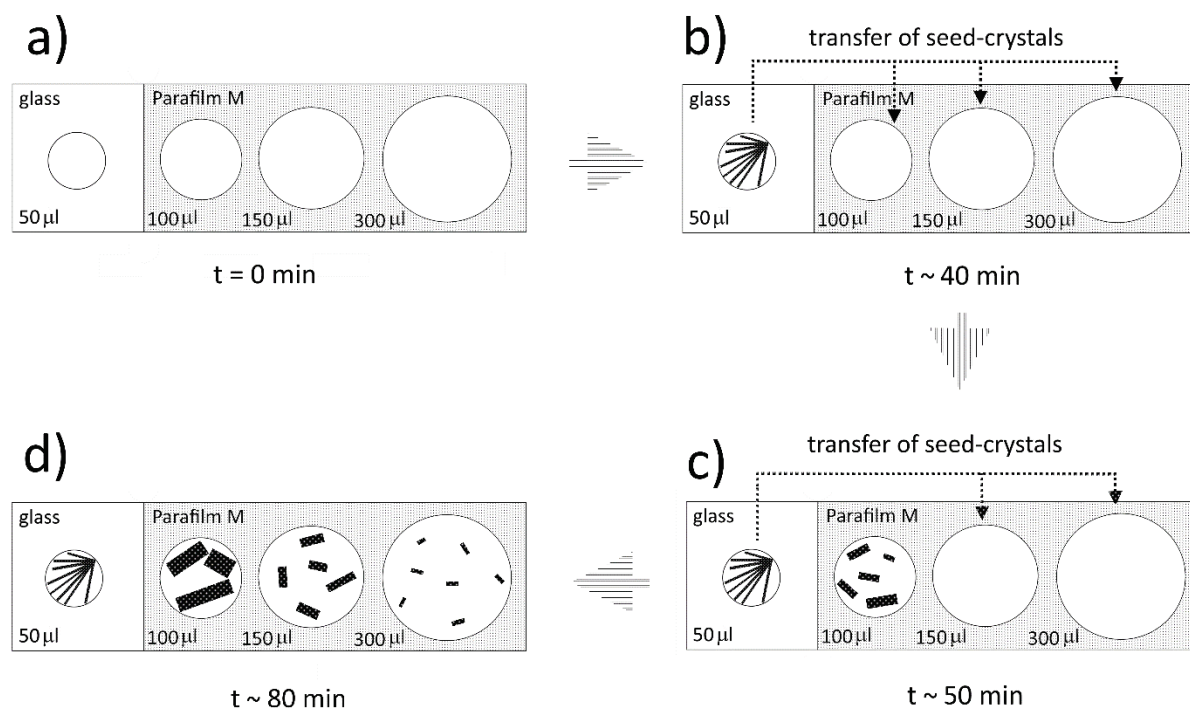


Figure S2 Scheme for the lab work 6b. a) Slide glass partly covered by Parafilm® M with 4 droplets (50 µl, 100 µl, 150 µl and 300 µl); b) Growth of seed-crystals in droplet of 50 µl and their manual transfer to other drops; c) Start of co-crystal growth in the drop of 100 µl and additional transfer of seed-crystals to the 150 µl and 300 µl drops; d) Growth of co-crystals in all drops.

6c. Lysozyme

Main goal – to get some experience with the crystallization of biopolymers

Approximate time limit: 2 academic hours

Tasks:

1. Learn about different crystallization techniques of biopolymers
2. Learn the hanging drop technique
3. Examine protein crystals in the drops under a microscope in non-polarized and polarized light

Substances and materials:

1. Hen egg-white lysozyme
2. Distilled water
3. 4 M stock solutions of NaCl
4. 1 M NaAc/HAc buffer pH = 4.0 (NaAc – sodium acetate, HAc – acetic acid)

Equipment:

Laboratory coat, goggles, gloves, 24-well hanging drop crystallization plates, P10 and P1000-type micropipettes with tips, 12 siliconized glass slides, 15 ml falcon tubes, racks for 15 ml

falcon tubes, 1.5 ml Eppendorf tubes, racks for Eppendorf tubes, pH meter, balances, laboratory fridge, microscope with polarizer and analyzer, WACKER® SILICON PASTE P4 or analog.

Description of lab work

Procedures carried out by an assistant before the class starts. It is also possible to have this work performed by the most advanced students:

15 mg of lysozyme is placed into a 1.5 ml Eppendorf tube, and dissolved in 485 μ l of distilled water, thus making a 30 mg/ml lysozyme solution. Lysozyme solutions with concentrations of 50 mg/ml and 70 mg/ml should be also prepared. A 1 M NaAc/Hac stock buffer with pH = 4 is prepared by slowly adding 1 M NaAc solution to a 1 M Hac solution while stirring to reach pH = 4, ensuring that the pH is controlled during this process. The 4 M stock solutions of NaCl are prepared by dissolving in distilled water the corresponding amount of NaCl in a 500 ml volumetric flask. The lysozyme solutions are stored in the fridge.

Work done by students:

Students are divided into pairs and calculate the required volumes of 4 M NaCl stock solutions and 1 M NaAc/Hac buffer to get 2 ml precipitant solutions (i.e. NaCl in buffer solution) with NaCl concentrations of 1.1M, 1.2M, 1.3M, and 1.4M. A teacher checks the calculated ratios and then each student prepares the 2 ml solutions using P1000-type micropipettes. The teacher shows how to grease the edges of the crystallization vessel, and each student repeats this procedure.

A teacher and the students then fill the wells of a 24-well crystallization plate with the antisolvent solutions (500 μ l), so that wells A1-A3 are filled with the 1.1 M NaCl solution, wells B1-B3 are filled with 1.2 M NaCl solution, wells C1-C3 are filled with 1.3 M NaCl solution, and wells D1-D3 are filled with the 1.4 M NaCl solution (Fig. S3). Each time before changing the antisolvent solution, the nozzle of the pipette must be changed.

A 2 μ l droplet of the 30 mg/ml lysozyme solution is then put on the siliconized glass using a P10-type micropipette; the tip of the micropipette is removed afterwards (Fig.S4a). After that, 2 μ l of the antisolvent solution from well A1 are added to the lysozyme droplet (Fig.S4b). The glass with a 4 μ l droplet is turned over and pasted above the antisolvent solution placed in the A1 well using grease (Fig.S4c). This is first demonstrated by a teacher, and then reproduced by the students. As a result, droplets of the 15 mg/ml lysozyme (initial concentration of the 30 mg/ml twice diluted) solution hang above wells A1, B1, C1, D1 (Fig.S4b). The same sequence is carried out with lysozyme solutions of 50 and 70 mg/ml and then placed above A2-D2 and A3-D3 wells, respectively (Fig. S3).

Above these antisolvent solutions the lysozyme crystals grow in approximately 40 mins. Students should be warned that the vapor diffusion process does not occur in such a short time and crystallization is due to the amount of precipitator solution that was added to the

drop. In the case of other proteins (not lysozyme) crystallization experiment takes from several days to weeks.

The crystals are inspected under an optical microscope in non-polarized and polarized light. The number and quality of crystals obtained under different crystallization conditions are compared and discussed.

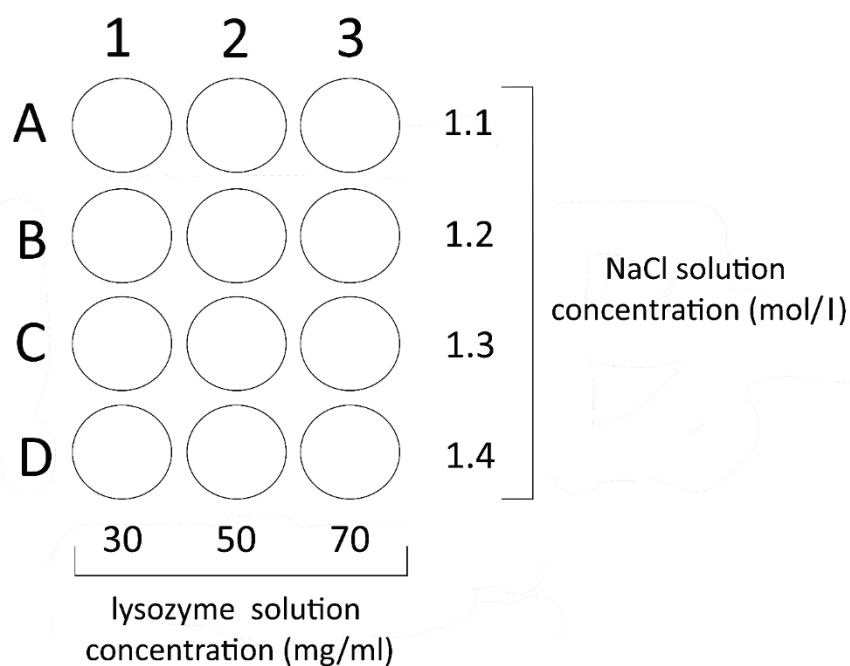


Fig. S3 Scheme of filling of a 24-well crystallization plate with a series of solutions

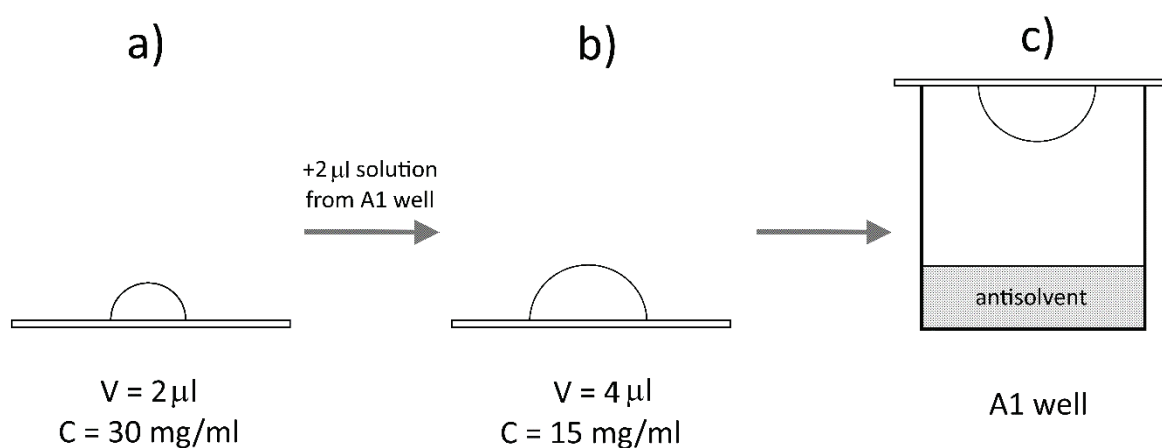


Fig. S4 The sequence of procedures to prepare a hanging drop crystallization set-up. a) Drop of lysozyme solution on siliconized glass; b) Twice diluted drop of lysozyme solution after addition of 2 μl antisolvent solution from A1 well; c) Hanging drop above antisolvent solution (A1 well)

Appendix 7. Samples of crystals grown by students

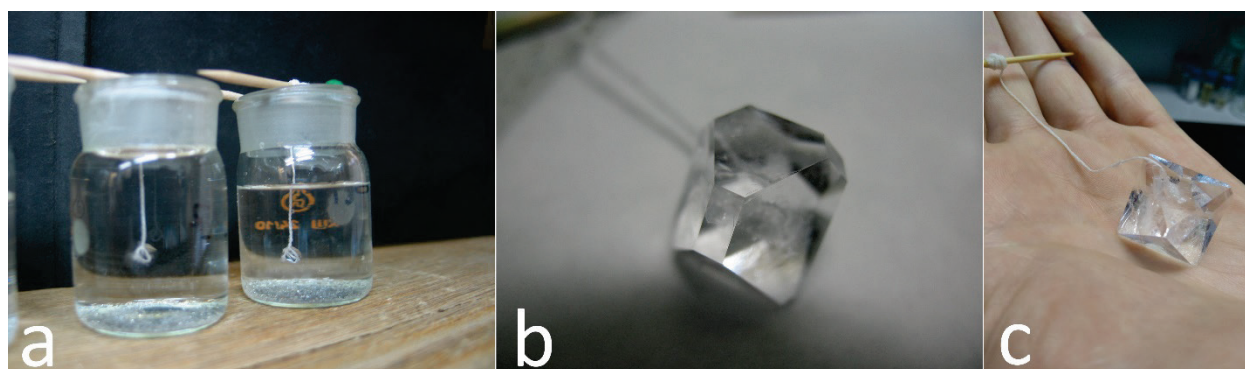


Figure S1. Potassium alum crystals grown by slow evaporation using seed crystals

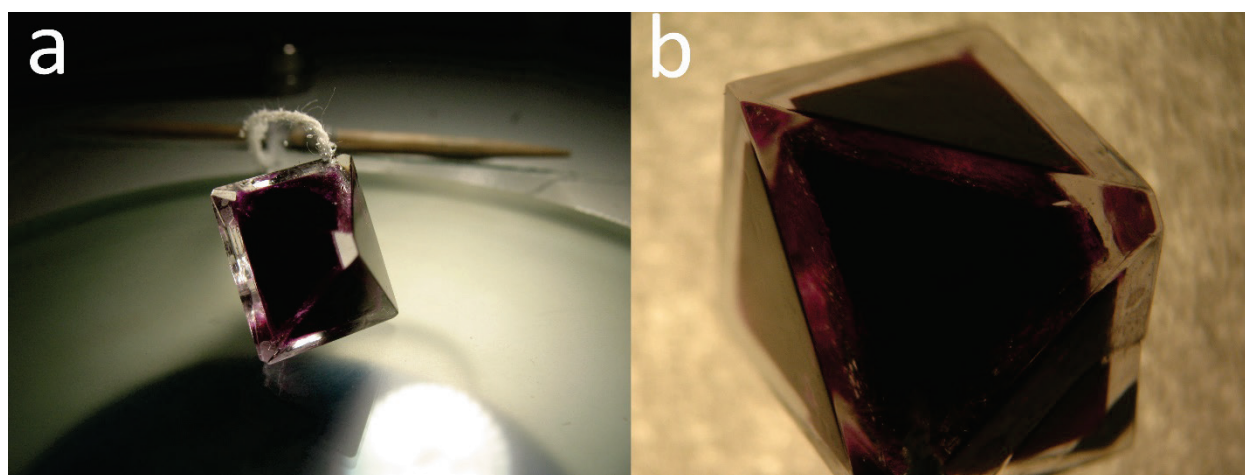


Figure S5. Growth of layered alum crystals. Chrome alum core (inside) covered by potassium alum shell (outside).

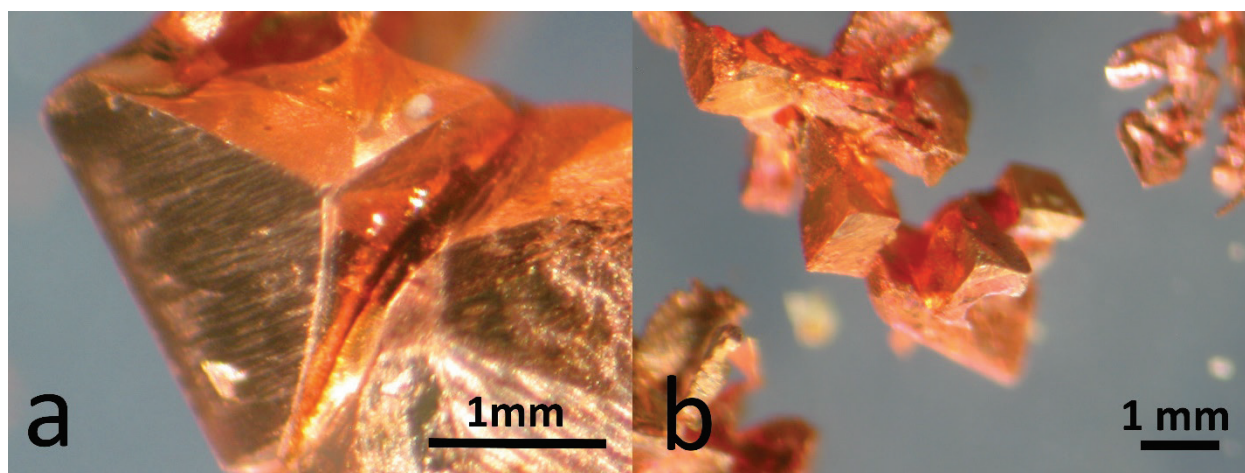


Figure S6. Copper crystals grown by redox reaction between CuSO_4 and Fe

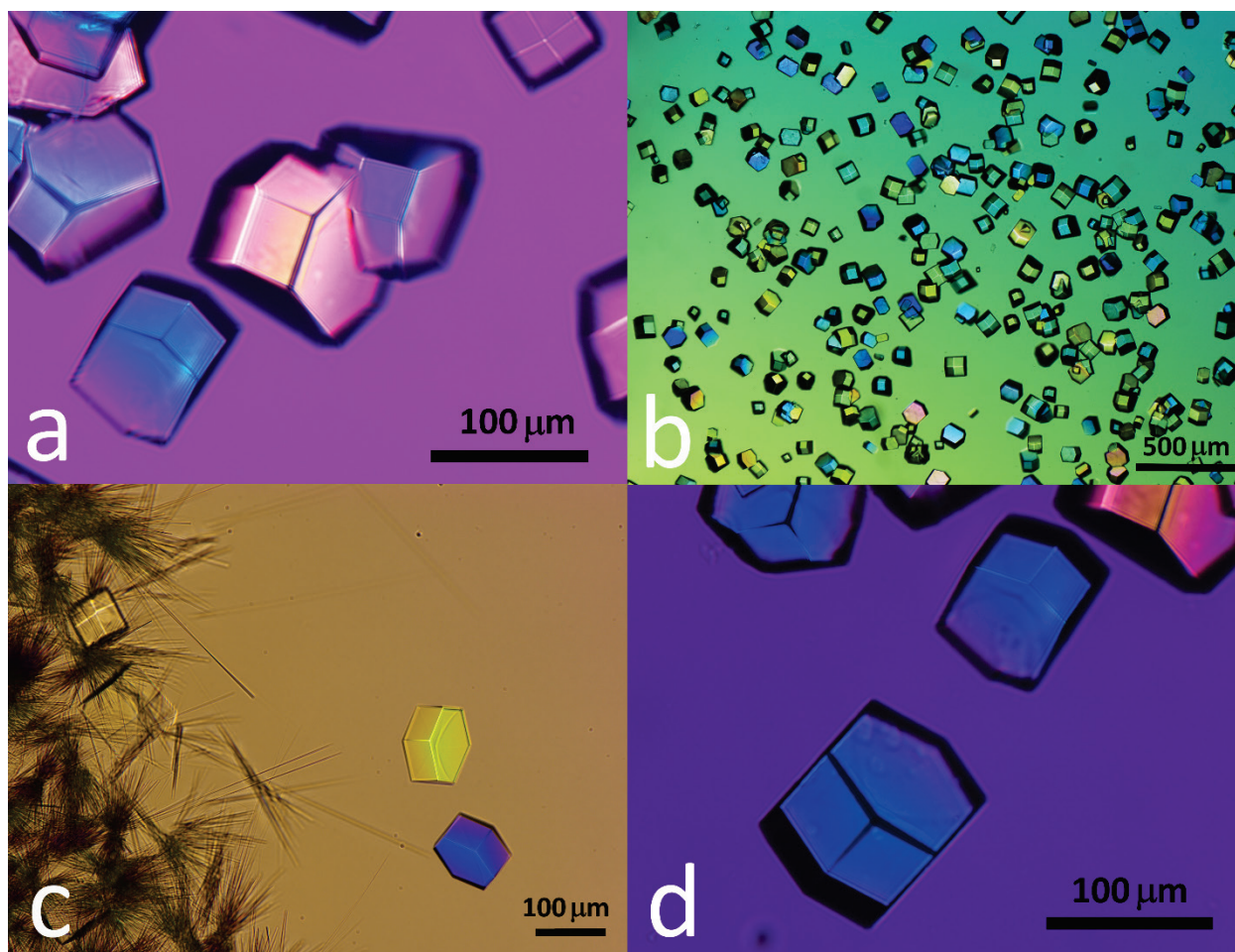


Figure S7. Lysozyme crystals under polarized light crystallized by hanging-drop vapor diffusion antisolvent technique.

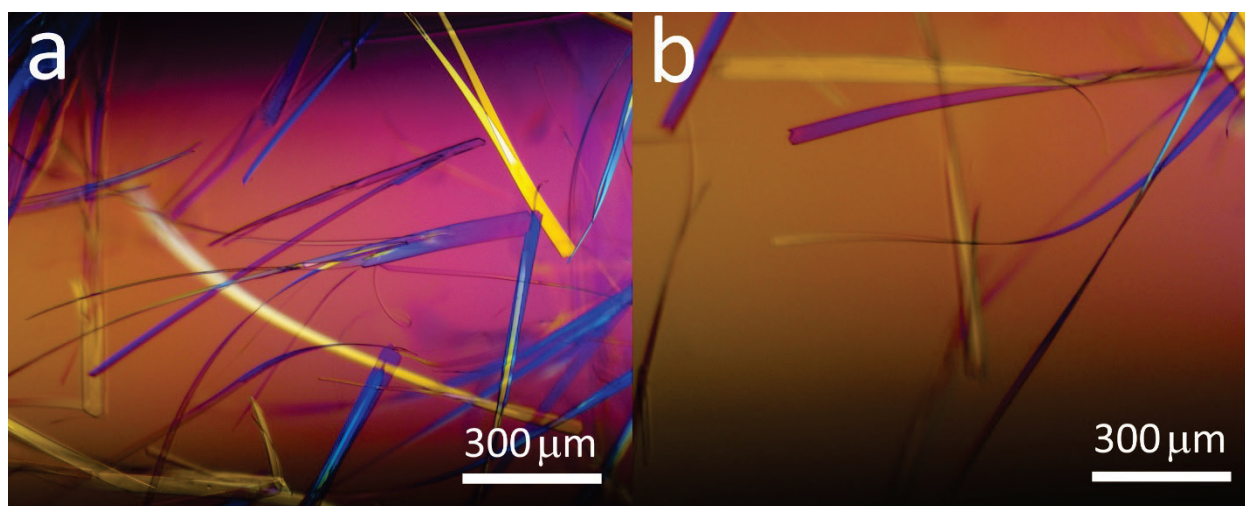


Figure S8. L-isoleucine single crystals having plastic mechanical properties were grown inside glass capillary by layering diffuse antisolvent crystallization. Optical microscopy under polarized light.

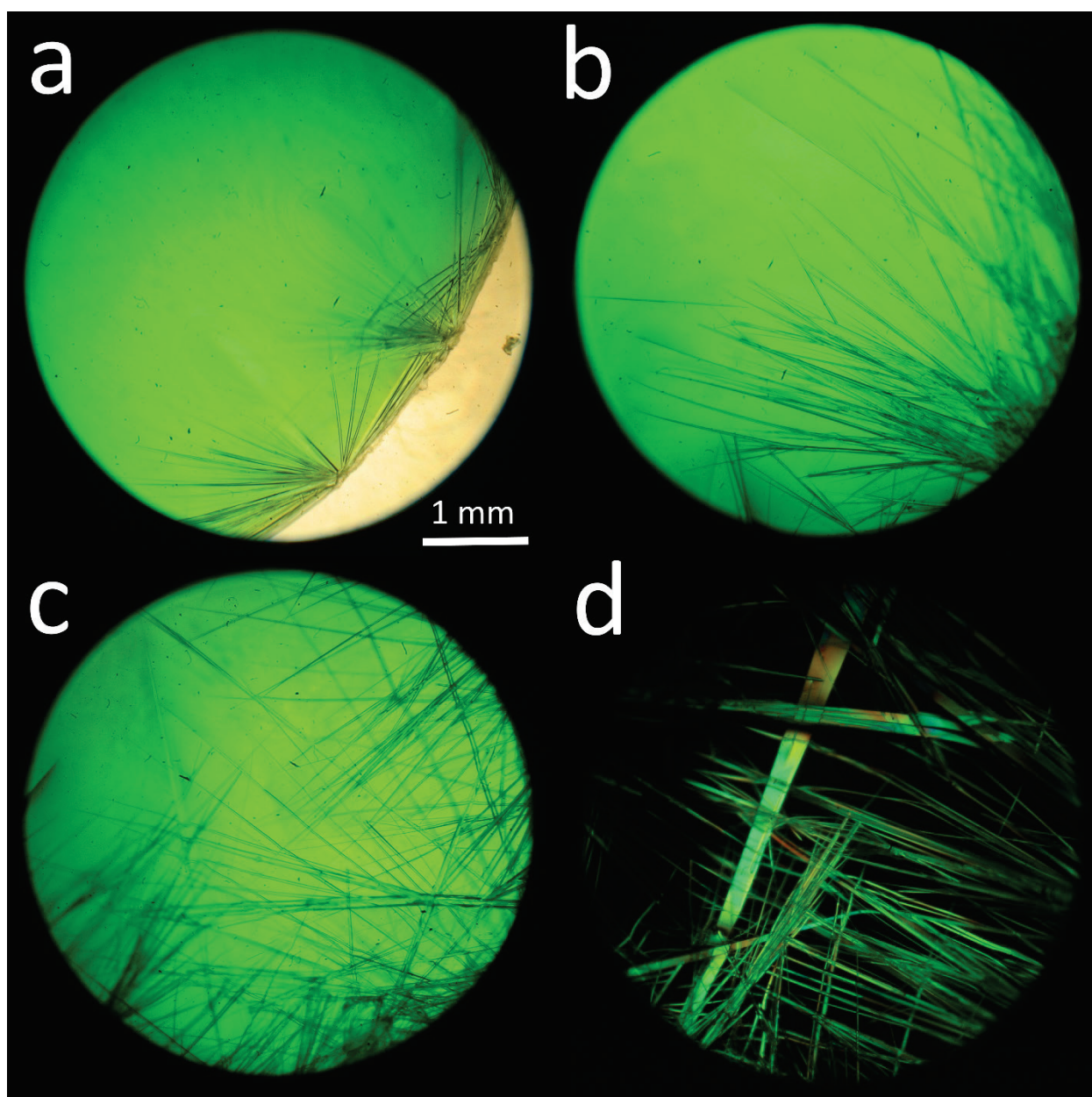


Figure S9. Growth of copper chloride dihydrate at different stages (slow evaporation crystallization from drop). Image (d) obtained using polarized light.



Figure S10. Sodium chloride crystals obtained by slow evaporation crystallization. a) Standard growth of cubic crystals from pure water solution and b) dendritic growth from water solution with addition of $\text{K}_4[\text{Fe}(\text{CN})_6]$

Appendix 8. List of equipment used when teaching the course

The lectures do not require any special equipment. In principle, even a chalk and a black-board would be sufficient. We use a computer and a projector for lectures, internet connection and one of the numerous programs that became so popular during the Covid restrictions, that make it possible to give and receive lectures also in a hybrid or a completely remote mode.

The laboratory exercises do not require sophisticated equipment for the basic part 1, and use the equipment of the research laboratories for part 2.

Laboratory exercises are carried out in a special room (18 m², laboratory tables, scales, fridge, distiller, fume hood, microscopes, individual protection means (laboratory coats, goggles, gloves), reagent storage cabinet, heating plates, ultrasonic bath, glass cups, glass funnels, filter paper, Petri dish, spatulas, toothpick, thread, sandpaper, filter nozzles for a syringe (pore diameter of 0.45 microns or 0.22 microns), pipettes, crystal manipulation kit, cryo-oil, markers.

Crystallization mushroom (Triana Sci&Tech®) (Garcia-Ruiz et al., 2002)

24-well plate (Molecular dimensions®)

Glass tubes for layering diffuse antisolvent crystallization

For advanced level experiments we use:

– A ‘Discoverer-1500’ DIA-type apparatus with a nominal force of 1500 tons at the Sobolev Institute of Geology and Mineralogy SB RAS in Novosibirsk, Russia. Eight “Fujilloy N-05” 26 mm tungsten carbide cubes with 12 mm truncation are used as inner-stage anvils. Pyrophyllite gaskets, 4.0 mm in both width and thickness, are used to seal the compressed volume and support the anvil flanks.

Parameters of the experiments: pressure up to 6 GPa, temperature up to 1500 °C.

– Precision heating furnace (± 0.1 °C, Eurotherm 2604, RIF, BS) for solid-phase synthesis, , spontaneous crystallization on a platinum loop and crystal growth by the modified Czochralski technique (top-seeded solution growth), mechanisms for rotation and translational movement of grown crystals. Operating temperatures up to 1150 °C (Sobolev Institute of Geology and Mineralogy SB RAS in Novosibirsk, Russia).

– Muffle furnace THERMOCERAMICS-1600 with operating temperatures up to 1600 °C for solid phase synthesis (Sobolev Institute of Geology and Mineralogy SB RAS in Novosibirsk, Russia).

- Diamond anvil cells of different types.

- Spray-drying (Mini Spray Dryer Buchi B-290)

- A laboratory-scale freeze dryer (one processing shelf, 250 × 350 mm, with a temperature range of –30 to +80 °C) (NIIC SB RAS, Russia) described in (Ogienko, A. G., Bogdanova, E. G., Trofimov, N. A., Myz, S. A., Ogienko, A. A., Kolesov, B. A., Yunoshev, A. S., Zubikov, N. V., Manakov, A. Yu., Boldyrev, V. V., & Boldyreva, E. V. (2017). Large porous particles for respiratory drug delivery. Glycine-based formulations. European Journal of Pharmaceutical Sciences, 110, 148-156)

- Mosquito Xtal3 robot for creating protein crystallization drop set

- Vibrational ball mill (Narva®, DDR)

- Vibrational ball mill Retzsch
- Home-made model devices for different types of mechanical treatment (see Tumanov, I. A., Achkasov, A. F., Boldyreva, E. V., & Boldyrev, V. V. (2011). Following the products of mechanochemical synthesis step by step. CrystEngComm, 13(7), 2213-2216; Losev E., Arkhipov S., Kolybalov D., Mineev A., Ogienko A., Boldyreva E., Boldyrev V. Substituting Steel for a Polymer in a Jar for Ball Milling does Matter, CrystEngComm. 2022.N9. P.1700-1703. DOI: 10.1039/D1CE01703A)