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**UNIVERSIDADE TÉCNICA DE LISBOA  
INSTITUTO SUPERIOR TÉCNICO:**

**Departamento de Eng. Química  
Centro de Química Estrutural  
Núcleo de Audiovisuais  
Gabinete de Apoio à Pós-Graduação:**

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# **ON THE ROCKS... CRYSTALLIZATION ON ROUGH SURFACES**

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Author: Clementina Teixeira, Assistant Professor at IST**

## **“On The Rocks...” Crystallization on Rough Surfaces**

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Maria José Rodrigues (high school teacher)

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**Music:** Johan Sebastian Bach, Brandenburg Concerto N° 3 in G Major (BWV 1048), Classical Collection, Parry Music, Inc.

**Produced by:** Núcleo de Audiovisuais, IST

**Text:** Dr. Clementina Teixeira

**Photographs:** Some photographs are reprinted, with permission, from “O Livro das Pedras”, 1996, by C. Teixeira. Most of them were taken by Eng. Marques Dias (IST). Some others are reprinted from the video.

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Some of the samples shown in this video were prepared by high school teachers, during a 50-hour intensive course on "synthetic minerals" (PRODEP II-FOCO, 1996).

### **Acknowledgements:**

We would like to thank all those who joined us in our enthusiasm for crystals, for their comments and suggestions. There were a gratifyingly large number of them. Profound thanks also to Núcleo de Audiovisuais, IST and to the high school teachers and students from Escolas Secundárias Manuel Cargaleiro, Palmela, Alfredo dos Reis Silveira and Padre Alberto Neto, our co-workers in Ciência Viva 96/97 - their trust gave us great confidence. This version was updated in June, 2000.

Instituto Superior Técnico, October 1997, June 2000



## ***“On The Rocks...”***

### ***A short introduction***

*On the threshold of the twenty-first century, we live surrounded by crystals. Natural gems and minerals that have seduced humankind throughout the ages have kept their importance, but crystals produced to make our lives easier and comfortable have become essential. Therefore, crystallization should no longer be regarded merely as a purification technique in our chemistry teaching programs: it has become an important tool in materials technology.*

*For the last four years, our research group at Instituto Superior Técnico, Centro de Química Estrutural, has investigated crystal growth on rocks and rough surfaces. Since 1996, several intensive 50-hour courses for high school teachers (a total of 200 attending) have been held to spread the most important information. The FOCO Program (Ministry of Education, European Community funds) finances these courses.*

*Supported by the Ministry of Science and Technology, within the Ciência Viva Program, many of these teachers were able to perform many experiments with their students in their regular laboratory programs of Chemistry, Physics and Geology, or at their “Science Clubs” in schools. Some of these results have been successfully presented at Science and Technology Exhibitions and Conferences but, apparently, this approach has not yet been used for teaching purposes. The co-operative interaction between both Ministries, the schools, and universities is very promising: new ideas are developing and immediately being applied, and joint research projects are emerging to solve specific questions and interesting problems arising from the application of our methodology. With an appropriate selection of both substances and techniques, this program can*

*be used in courses from the basic level in primary schools up to university. We are interested in finding partners in other countries who might share our program of research, and this invitation is also extended to companies working in the development of teaching materials.*



*Figure 1. Potassium alum  $KAl(SO_4)_2 \cdot 12H_2O$ , showing a tabular habit. A piece of quartz is the host. This picture became the “ex-libris” of our project. A first year student of Laboratories I, of the Chemical Engineering course at Instituto Superior Técnico (eighteen years old) prepared the sample. A different version of this photograph was published by Teixeira, C.: *Os Cristais no Ensino e Divulgação da Química, Colóquio Ciências 2000, Fundação Calouste Gulbenkian*, 25, 20-36.*



## **1. Introduction - Crystallization and Crystal Growth**

In the early days of chemistry, crystallization was often the only experimental method for purifying substances. The development of chromatography and other separation techniques reduced its importance to some extent, which is why it is covered only briefly in most modern general chemistry textbooks. However, observing the world around us, and not even considering the enormous variety of natural crystals studied in mineralogy and geology, we realize that crystals have in recent decades become an important part of daily life: sugar and salt in our food; aspirin, vitamin tablets and other medicines; pretty artificial jewelry for parties; synthetic diamonds for cutting tools used by surgeons and in modern machinery; rubies for lasers; sophisticated optics in many instruments; silicon chips in electronic cards; liquid crystals in our computers and calculators, etc. We finally realize that in modern technology, crystallization, together with crystal growth, are much more important than we were told at school! It is therefore important that, whenever this subject is introduced in any field of science (physics, chemistry, mineralogy and geology, material sciences) a summary of its major applications be presented:

**a) Crystallization** is still a classical laboratory technique for separation and purification of substances and an important tool in studies of natural crystals in mineralogy and geology; it has become an important industrial technique for large-scale production of small crystals.

**b) Crystal growth** is applied mainly in two fields: as a complement to the methods of synthesis for the characterization of compounds, with the production of small single crystals for X-ray diffraction methods and in materials technology for the production of large single crystals.

A more detailed description by Hulliger (1), in decreasing order of importance in chemical technology, is shown in Box 1.

## **Box 1**

### **Crystallization and Crystal Growth in the field of Chemistry**

- **Industrial crystallization:**  
production of polycrystalline bulk materials (NaCl, sugar, urea, zeolites)  
production of silicon single crystals
- **Structural analysis:** production of small single crystals (less than one mm)
- **Separation, purification, and characterization of chemicals and natural products, including racemate separations**
- **Crystallization of macromolecules (proteins, polymers)**
- **Measurement of physical properties**
- **Biocrystallization in the medical field**
- **Synthesis of catalysts (zeolites)**

Notwithstanding all the high-tech methods available nowadays for “crystal engineering”, one can introduce crystallization in an attractive and simple way, using inexpensive equipment or even at home, with common glassware available in any kitchen, **as long as strict safety rules are followed.** Among compounds producing large single crystals (mainly water-soluble ionic metal salts), a large number can crystallize from supersaturated solutions on rough surfaces like those of rocks and shells. The beauty of the crystals produced, as well as the diversity of results obtained for each substance, when rocks of different chemical compositions are used, are a source of endless ideas for a teacher to introduce many basic concepts in chemistry and other sciences where crystals and solid state are important topics of study. In the following sections, a summary of this method is given. More detailed information is available in the literature (2-7).



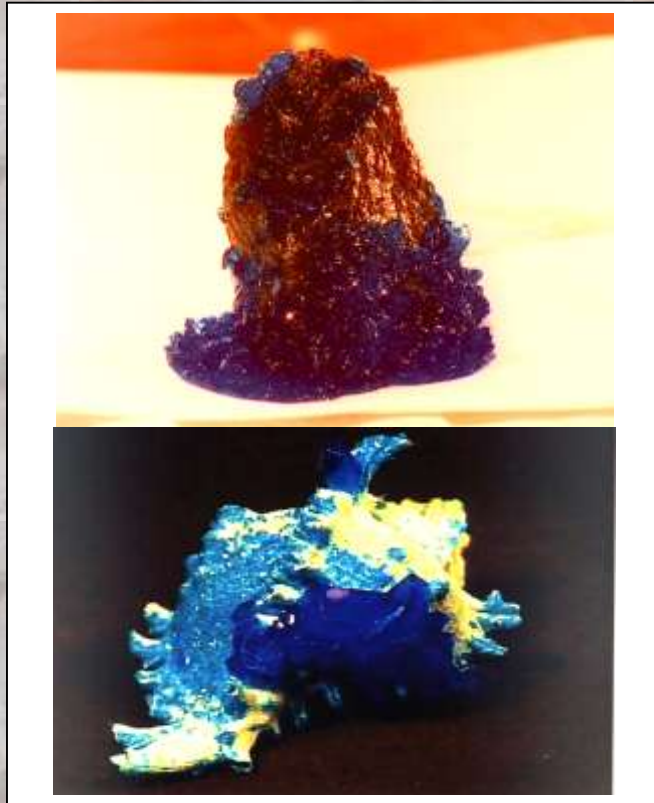


Figure 2. Hydrated copper sulfate,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , is one of the best substances to start crystal growth on rough surfaces. The results are practically guaranteed for any material used as host. Strong reactions are liable to occur with certain types of hosts, as discussed in the text (2, 6, 7).

## **2. Some details of the method**

There are four major subjects to consider in this method, as illustrated in Box 2:

- The best substances to use as solutes**
- The materials used to stimulate crystal growth**
- The reactions that can be developed between these two groups of substances**
- The best techniques for crystal growth**



## **Box 2**

### **SUBSTANCES**

Elements: S<sub>8</sub>, Cu, Ag, Pb, Sn

**COMPOUNDS:** Inorganic Ionic Salts

Organic



### **REACTIONS or OTHER INTERACTIONS**



### **HOST MATERIALS**

(rough surfaces)

Metal wires, pipe cleaners, tissues, wood

glass

**ROCKS and MINERALS**

**SHELLS**

**CRYSTALLIZATION TECHNIQUE IN SOLUTION:**

**COOLING/EVAPORATION OF SOLVENT**

Solvents - **Water**, others

## **2.1 Classification of Substances for Crystal Growth**

The main classes of substances are given in Box 3. The samples shown in the video are listed in Table 1, by order of appearance, with some remarks for each substance, including the crystal system, and which level of studies the preparation should be recommended for. Many substances are suitable for use in primary schools, whereas others can be synthesized only at the university level. Of course a simple non-toxic substance for use in primary schools can also be suitable for studies at university. It will mainly depend on the difficulty of the problems involved.



Figure 3. Potassium sodium tartrate  $\text{KNa}(\text{C}_4\text{H}_4\text{O}_6) \cdot 4\text{H}_2\text{O}$ , Rochelle's salt (2). A volcanic rock was used as host. This is one of the easiest and least toxic preparations, suitable for primary school work. The crystals are well worth studying: one of their interesting properties is piezoelectricity. This compound, derived from tartaric acid was involved in the studies by Louis Pasteur on crystallography and fermentation processes (3, 6g).

### **Box 3**

#### **Classification of Crystalline Substances**

**a. Metals**

**b. Molecular crystals (organic compounds, elements, etc.)**

**c. Anhydrous ionic salts**

**d. Hydrated ionic salts**

**e. Hydrated ionic double salts**

**alums  $M^I M^{III}(SO_4)_2 \cdot 12H_2O$**

**schönites  $M_2^I M^{II}(SO_4)_2 \cdot 6H_2O$**

**f. Solid solutions (co-crystallization)**

**g. Heterogeneous mixtures (with more than one phase of crystallized material)h. Other complexes (including organometallic compounds)**

Except for schönites (Figure 4), some of these substances are well known for producing large single crystals, so original references to them in the literature are difficult to find. Some are cited in encyclopedias and in books on crystallography and crystallization (8-12). However, we should say that the main search for compounds in the literature was made using references 13 and 14, looking only for the keywords “crystals” and “large crystals”. This search was very useful in providing further information on the toxicity of these compounds. **(We cannot recommend in this text any references, no matter how good and interesting they may be, which do not include safety rules together with the preparations).** In combination with this classification of crystals, according to their type of bonding, we recommend the attractive way of presenting the structures of solids adapted from Fortman, reference 15 (Figures 5-7).



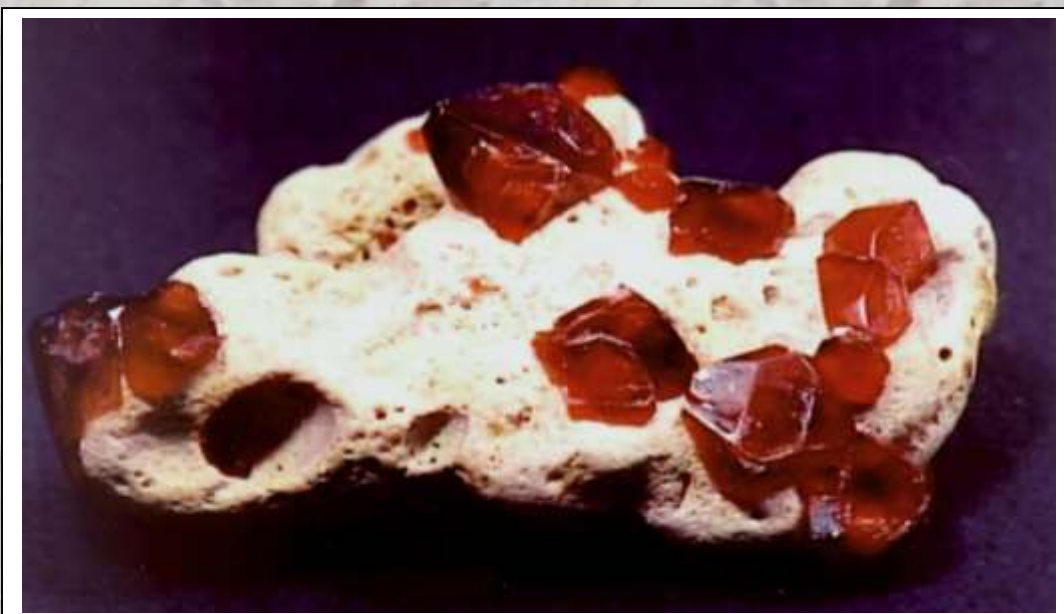


Figure 4. The very stable cobalt schönite:  $(\text{NH}_4)_2\text{Co}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ .

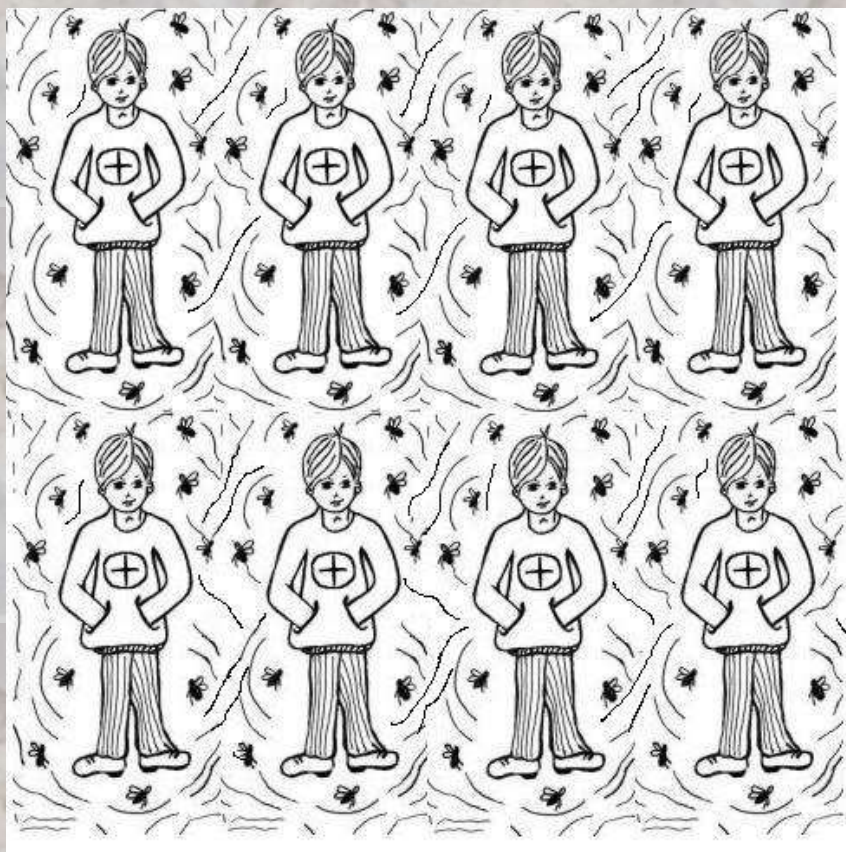


Figure 5. Pictorial analogy for the structure of metals, adapted from ref.15.

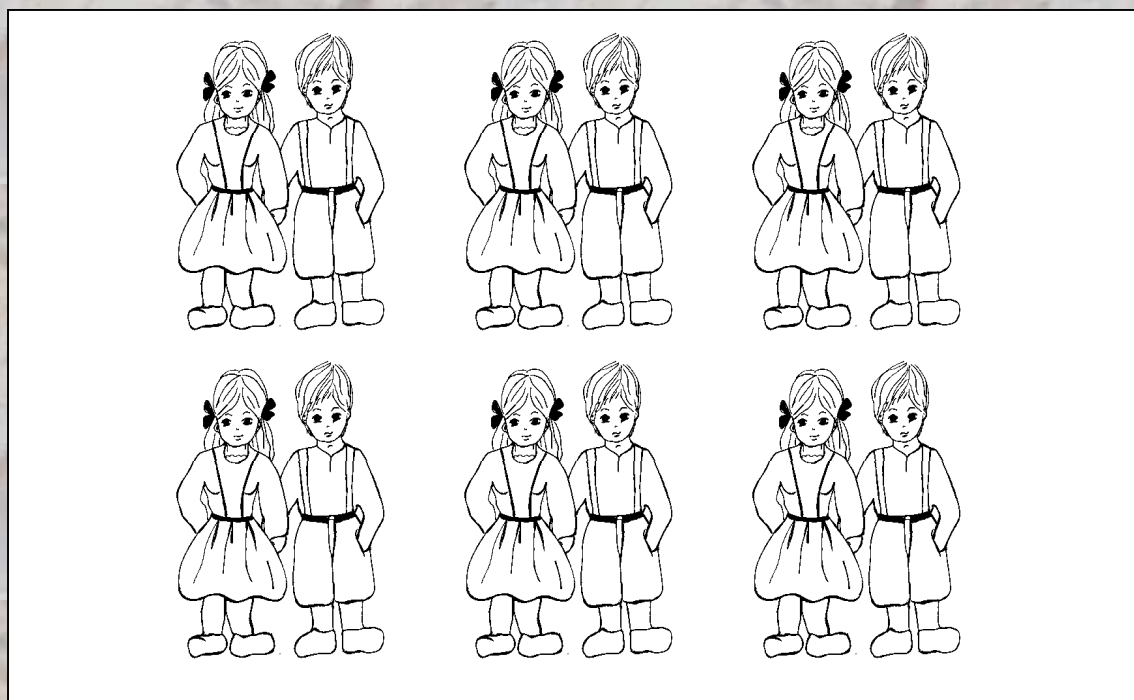


Figure 6. Pictorial analogy for the structure of molecular solids: each couple is a discrete molecule [15].

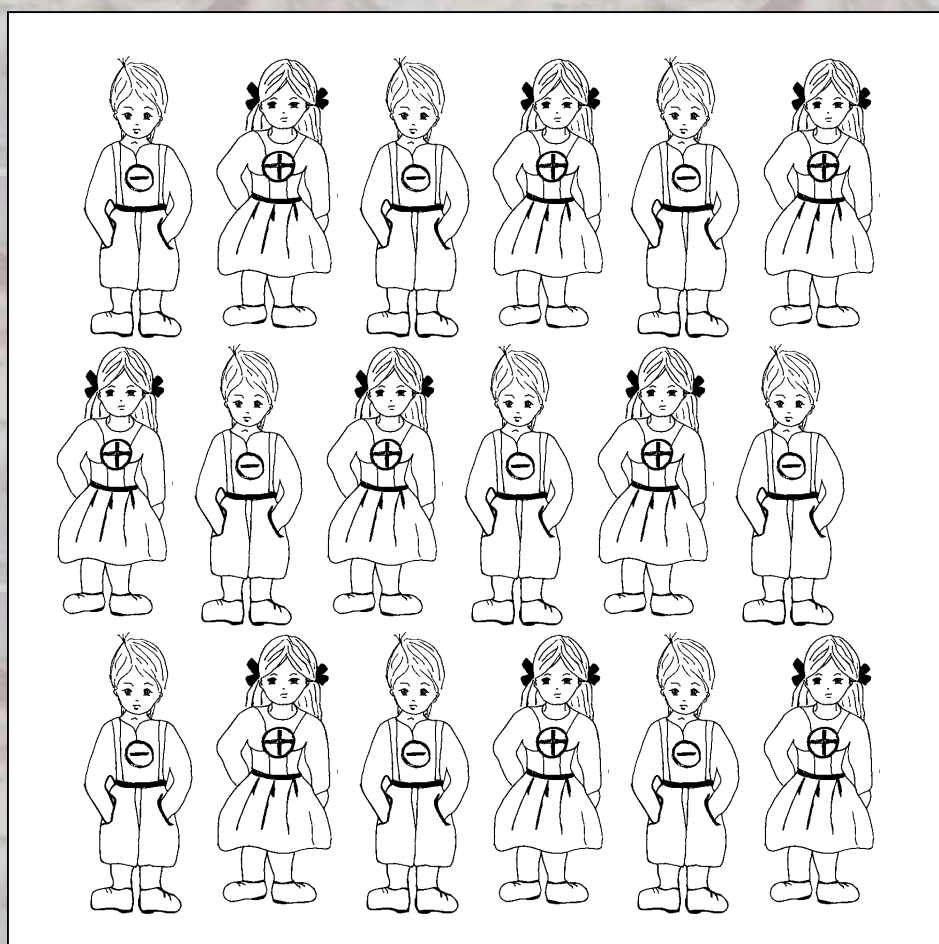




Figure 7. Pictorial analogy for the structure of anhydrous ionic solids: girls are cations and boys, anions [15].

Our search was mainly directed towards water-soluble inorganic compounds, for obvious reasons: water is a readily available solvent and such compounds will have a strong affinity for rocks, whose composition is mainly inorganic. However, any substance that grows large single crystals by traditional methods will be a potential candidate to end up “on the rocks”. Certain substances that might cause dangerous or undesirable reactions with rocks or shells should be avoided, as discussed in section 2.3. The complete list of solutes that we have tested is too extensive to include in this brief presentation. The recipes and characteristics for these and many other substances are given in reference 2 and others, cited therein.

Alums,  $M^I M^{III}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  (Figures 1, 8 and 9) and schönites,  $M_2^I M^{II}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  (Figure 4), are the best examples of isomorphous compounds, forming wonderful solid solutions in all



Figure 8. Ferric alum,  $(\text{NH}_4)\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  These crystals cannot be prepared by the traditional addition reaction between the single salts. This compound is unstable and starts to decompose after a few months (2, 7). Reprinted from Teixeira, C. *O Livro das Pedras* 1996, IST (2).



proportions ( $M^I$  is an alkali metal or monovalent ion like ammonium;  $M^{II}$  and  $M^{III}$  are metals with +2 and +3 valences). While the first family is fairly well known (11, 12, 16), we found only scattered information on the schönites family in analytical and structural chemistry books (9, 11, 12). We adopted the nomenclature in reference 12: as the alum family is called after its most popular compound, potassium alum (Figure 1), the schönites inherited their name from the mineral  $K_2Mg(SO_4)_2 \cdot 6H_2O$ , which has this designation. All these double sulfates (with the exception of ferric alum, Figure 8) can be prepared by reaction of the stoichiometric amounts of the corresponding single salts. Some of them are also commercially available as analytical reagents and for industrial use (chromium alum, Figure 9, and a few more alums; ammonium ferrous sulfate, Mohr's salt  $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$ , etc.).



Figure 9. Chromium alum,  $KCr(SO_4)_2 \cdot 12H_2O$ . These crystals are efflorescent. It is possible to keep a sample for a year or even more, if the sample is kept in the presence of some moisture, and away from bright light and heat. Once decomposed, it should be thrown away immediately (2).

The synthesis of potassium alum (Fig. 1) from aluminum, described by Summerlin (17, 18), combined with this method of crystallization, has become increasingly popular, as well as the characterization of the compound and the preparation of solid solutions (Figure 10)(2, 3, 5a1).



Figure 10. Solid solutions (mixed crystals) of potassium and chromium alums,  $\{KCr(SO_4)_2 \cdot 12H_2O, KAl(SO_4)_2 \cdot 12H_2O\}$ . Called “Amethysts” or “Fluorites” on the basis of their resemblance to the natural gems (2).

**Any work with nickel, a proven human carcinogen, should be done in a laboratory with proper ventilation and great care, or should be avoided as student work and only shown on video. Cobalt compounds are also toxic and should be handled with great caution (Figures 4, 11-15).**



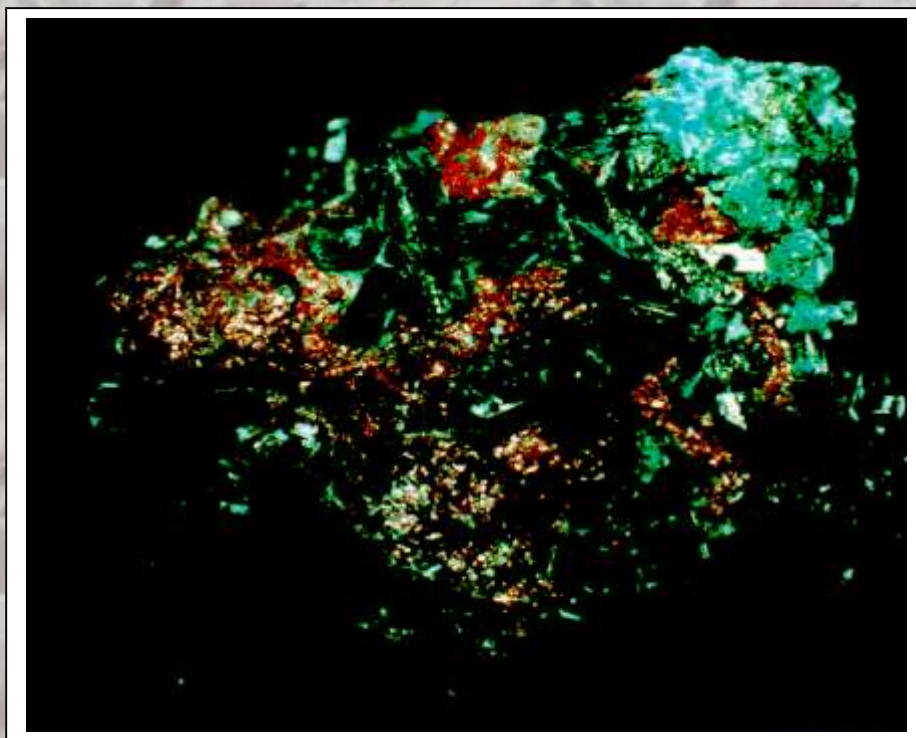


Figure 11. Two samples of nickel sulfate heptahydrate,  $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$  (2). This compound is strongly efflorescent: a fresh, non-dehydrated sample is shown at the top of this figure; the one below is already decomposed. It is possible, however, to preserve the shape of the crystals by protecting them with a thick layer of transparent nail polish. The preparation is described in references 2 and 10. Reprinted from Teixeira, C. *O Livro das Pedras* 1996, IST (2).



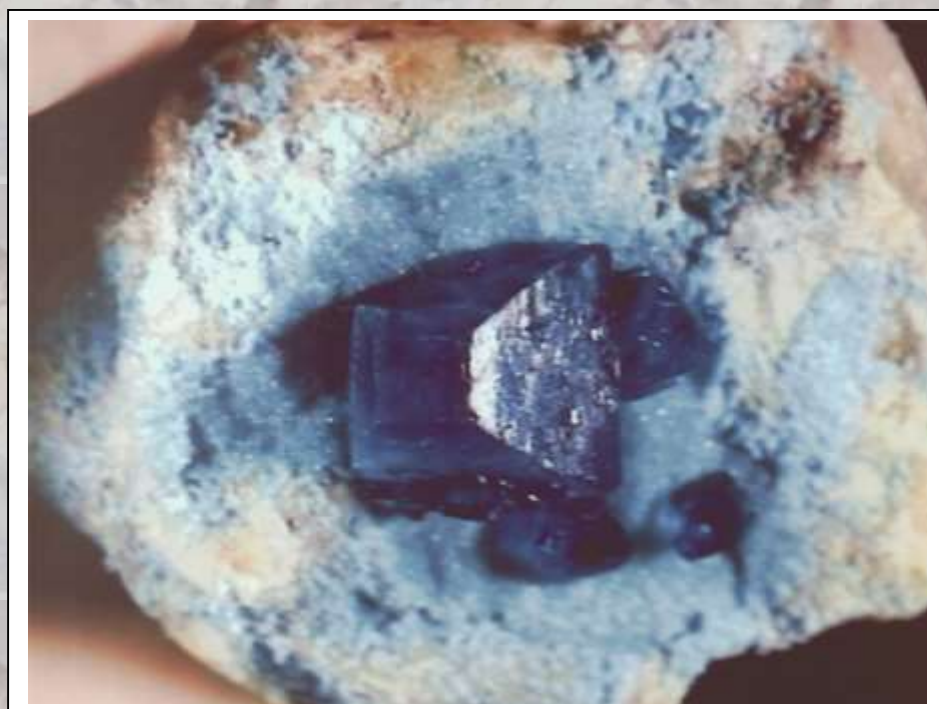


Figure 12. Nickel sulfate hexahydrate,  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$  (2). This is much more stable than the heptahydrate. Its distinctive bluish color and chunky crystals are readily recognizable. The preparation is described in references 2 and 10.



Figure 13. The bluish-green efflorescent solid solutions of copper and nickel sulfates. The crystal system changes with the proportions of the two components (2).

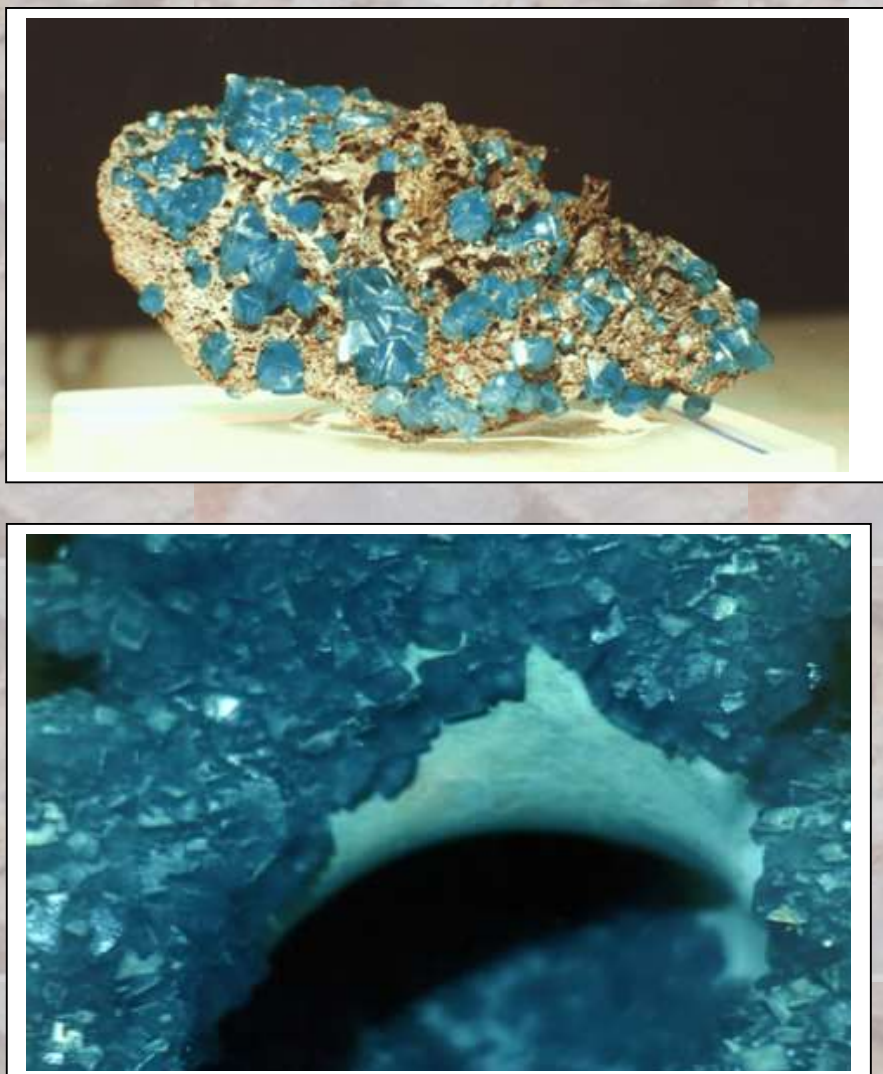


Figure 14. The bluish-green, very stable, nickel schönites:  $(\text{NH}_4)_2\text{Ni}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ , ammonium schönite, is shown at the top;  $\text{K}_2\text{Ni}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ , below, has a slightly stronger bluish color (2).

Solid solutions (mixed crystals) of schönites with low nickel or cobalt contents, diluted with magnesium or zinc, are the best way to keep crystals colorful and to reduce the toxicity of the preparations (Figures 16 and 17). The details for preparing a wide variety of these and other solid solutions, as well as heterogeneous mixtures, together with the recipes and properties of each compound, solubility data and many other suggestions, are included in references 2 and 3. Some



compounds will be highly stable (potassium ferricyanide, Figure 18). Others, like practically all iron salts, will slowly decompose (Figure 8).



Figure 15. A solid solution of cobalt and nickel ammonium schönites (2),  $(\text{NH}_4)_2\text{Ni}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  and  $(\text{NH}_4)_2\text{Co}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ , prepared from a solution containing equal molar amounts of the two compounds. The sample is green, as the content of nickel schönite, the less soluble compound, is higher in the solid state; conversely, the solution is brown, with a higher content of the cobalt compound at the end of the crystallization process.

Some efflorescent salts will be better preserved if kept in a flask containing some moisture (Figure 9). “Amethysts”, the solid solutions of chromium and aluminum alums, are stabilized by this last compound and do not retain the efflorescent character of chromium alum (2) (Fig. 10). The formation of metallic crystals is always associated with oxidation-reduction reactions, and was never tried on the rocks. A brief discussion is included in section 2.3.



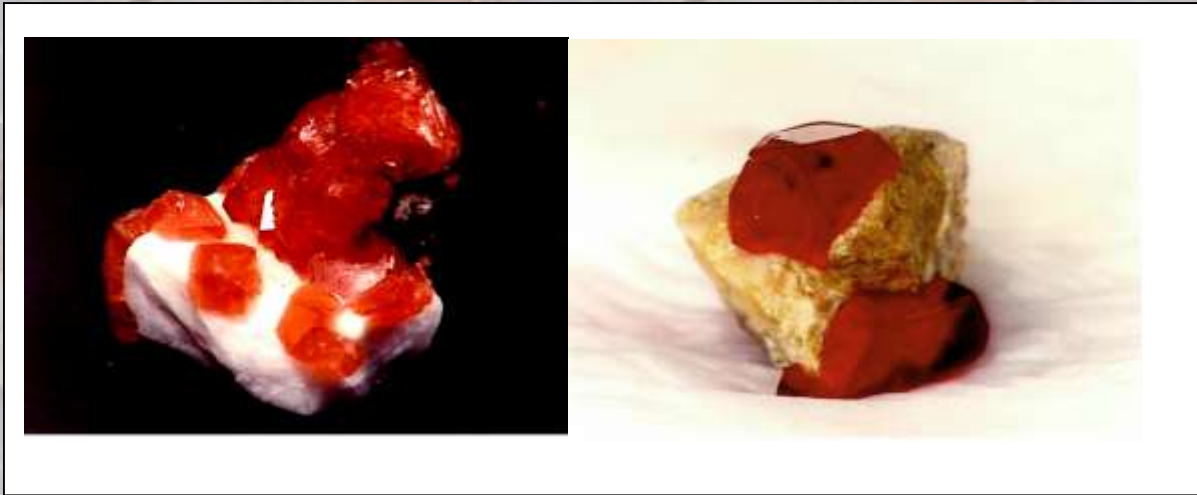


Figure 16. Solid solutions of cobalt, zinc and magnesium schönites (2): a way to preserve the color and reduce toxicity.

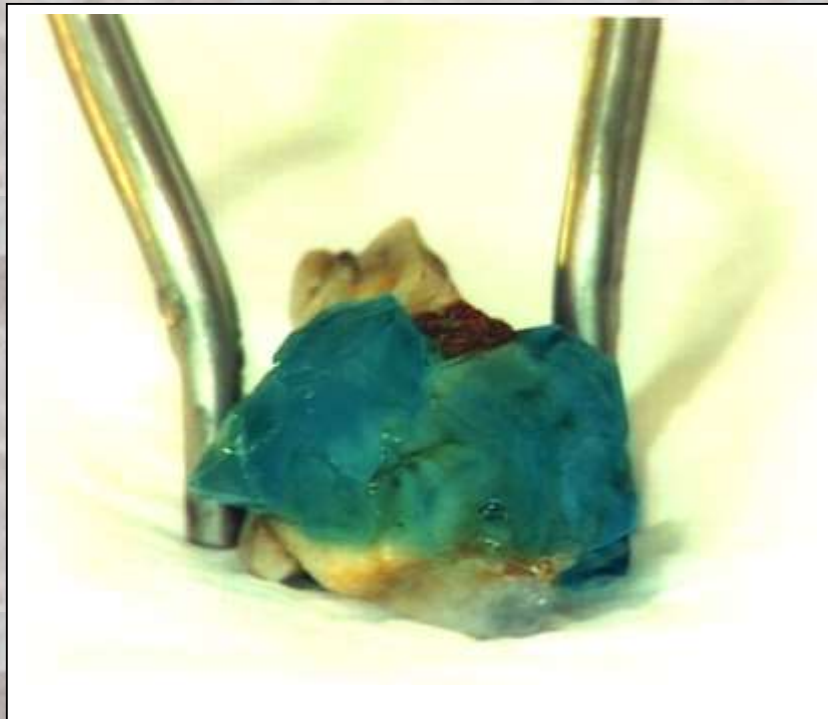







Figure 17. Solid solutions of nickel, zinc and magnesium schönites (2).

**Table 1**



**List of substances included in the video, in order of appearance.**

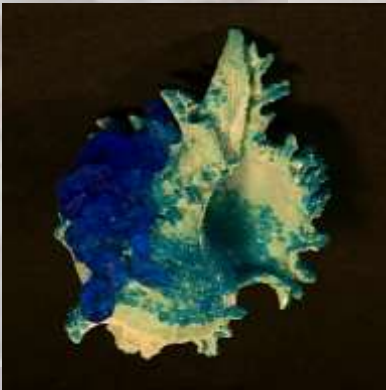

All the preparations and safety rules are included in ref. 2 and references cited therein.

Order	Classification of Substances	Crystal System	Level of Studies	Observations
<b>Molecular Crystals</b>				
1	Sulfur, S <sub>8</sub> 	Orthorhombic	University	Recrystallized from carbon disulfide on volcanic rocks; highly toxic solvent!
2	glycine, NH <sub>2</sub> CH <sub>2</sub> COOH 	Monoclinic	Primary school	

Anhydrous ionic salts				
3	$(\text{NH}_4)\text{H}_2\text{PO}_4$ 	Tetragonal	Primary school	Inclusion of dyes in transparent crystals (Ponceau 4R-red) (19)
4	$(\text{NH}_4)\text{H}_2\text{PO}_4$ 		Primary school	Inclusion of dyes (tartrazine-yellow) (19)
5	$(\text{NH}_4)\text{H}_2\text{PO}_4$ 		Primary school	A volcanic rock acts as a habit modifier: a saturated solution with whiskers



	$(\text{NH}_4)\text{H}_2\text{PO}_4$ 			<p>The same sample, removed from the solution</p>
6	$(\text{NH}_4)\text{H}_2\text{PO}_4$ 		<p>Primary school</p>	<p>Granite stimulates crystal growth</p>

Hydrated ionic salts				
7	$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 	Triclinic	Primary school	Extra precautions (6)
	Solution of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 			



The same rock before and  
after crystallization




Basalt


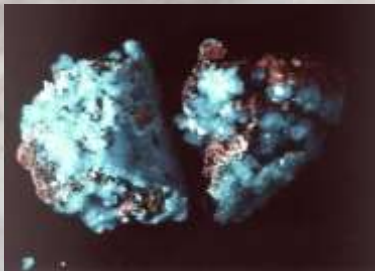
8	$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ 	Tetragonal	University	<b>Strict precautions or only for video presentation (6,10)</b>
9	$\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ 	Orthorhombic	University	<b>Strict precautions or only for video presentation</b> Efflorescent (10)



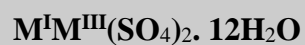
Solid Solutions of Hydrated Salts				
10	$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ 	The crystal system changes with composition	University	<b>Strict precautions or only for video presentation</b> Efflorescent (6)




Hydrated ionic double salts				
11	$\text{KNa}(\text{C}_4\text{H}_4\text{O}_6) \cdot 4\text{H}_2\text{O}$ 	Orthorhombic	Primary school	Rochelle salt (10)



12	$\text{KNa}(\text{C}_4\text{H}_4\text{O}_6) \cdot 4\text{H}_2\text{O}$	Orthorhombic	Secondary schools	Copper sulfate in the solution is adsorbed at the surface (10)
				
13	$(\text{NH}_4)_2\text{CuCl}_4 \cdot 2\text{H}_2\text{O}$	Tetragonal	Secondary schools	Marble should not be used as host rock ( 6)
				

### Hydrated ionic double salts- - Alums

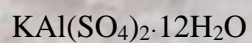
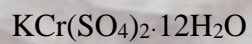


14	$KCr(SO_4)_2 \cdot 12H_2O$ 	Cubic	Secondary schools	Efflorescent (6)
14	$KCr(SO_4)_2 \cdot 12H_2O$ 	Cubic	Secondary schools	Partially decomposed by reaction with calcium carbonate in the shell (6)
15	$KAl(SO_4)_2 \cdot 12H_2O$ 	Cubic	Primary schools	With precautions (6,7)



### Solid Solutions of Alums

16






Cubic

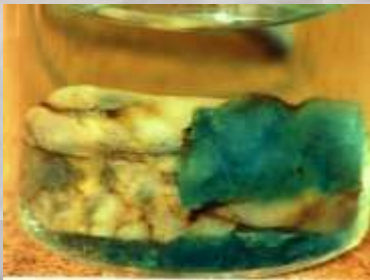

Secondary  
schools

With some shells there is a strong  
reaction and the products of  
decomposition can be observed (6, 7)

Hydrated ionic double salts Schönites $M_2^I M^{II}(SO_4)_2 \cdot 6H_2O$				
17	$(NH_4)_2Cu(SO_4)_2 \cdot 6H_2O$ 	Monoclinic	Primary school	Precautions (6, 7)
				
17	$(NH_4)_2Cu(SO_4)_2 \cdot 6H_2O$ $CuSO_4 \cdot 5H_2O$ 		Secondary schools	Two separate phases



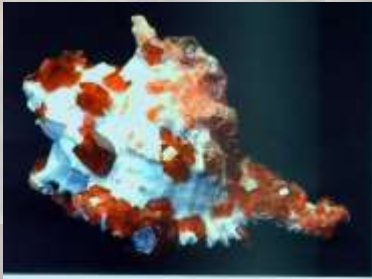

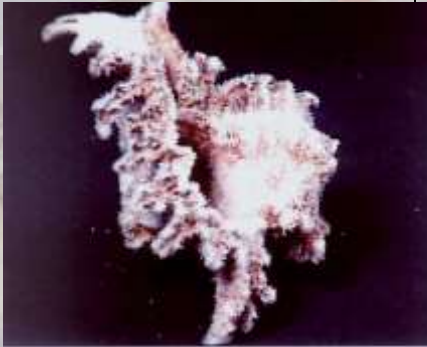
18	$(\text{NH}_4)_2\text{Mg}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	Monoclinic	Primary school	(7)
				
19	$\text{K}_2\text{Ni}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	Monoclinic	University	<b>Strict precautions or only for video demonstrations</b>
				
21	$(\text{NH}_4)_2\text{Co}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	Monoclinic	University	<b>Precautions (6)</b>
				

Solid Solutions of Schönites				
20	$(\text{NH}_4)_2\text{Ni}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Mg}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Zn}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ 	Monoclinic	Secondary Schools	<b>Precautions</b> In solution, very low contents of nickel are recommended (3)
22	$(\text{NH}_4)_2\text{Co}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Mg}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Zn}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ 	Monoclinic	Secondary Schools	<b>Precautions</b> Low levels of cobalt are recommended (3)



23	$(\text{NH}_4)_2\text{Co}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Mg}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Zn}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	Monoclinic	Secondary Schools	<b>Precautions</b> Low levels of cobalt are recommended (3)
				
24	$(\text{NH}_4)_2\text{Co}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Mg}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Zn}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	Monoclinic	Secondary Schools	<b>Precautions</b> Low levels of cobalt are recommended (3) Reaction with the rock
				

25	$(\text{NH}_4)_2\text{Ni}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Co}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Mg}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Zn}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ 	Monoclinic	University	<p>Rich in nickel</p> <p><b>Strict precautions or only for video demonstrations</b></p> <p>(3)</p>
26	$(\text{NH}_4)_2\text{Ni}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Co}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Mg}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Zn}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ 	Monoclinic	University	<p>Rich in Co</p> <p>with strong reaction</p> <p><b>Precautions (3)</b></p>

26	$(\text{NH}_4)_2\text{Ni}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Co}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Mg}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ $(\text{NH}_4)_2\text{Zn}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ 	Monoclinic	University	<p>Rich in Co</p> <p><b>Precautions (3)</b></p>
27	<p>several schönites, mixed crystals of unknown proportions</p>  			<p>with potassium reaction with the shell</p>



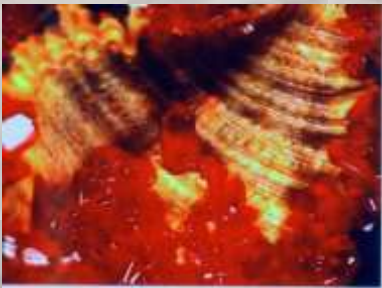
Other Complexes					
28	$\text{K}_3\text{Fe}(\text{CN})_6$ 	Monoclinic	Secondary schools	(6)	



Figure 18. Potassium ferricyanide. Very stable and easy to prepare, using any kind of host (2).

## 2.2 Host Materials

Let us now consider the best host materials, which **always have a rough surface**. Glass, pipe cleaners and metal wires are commonly used. Glass will not react with the solutions and very pure crystals will be formed. Copper wires are very popular for growing hydrated copper sulfate crystals, but very high concentrations are required (>80 g/100 ml water). Other metal wires are discussed in the next section. Rocks and shells are much less studied and therefore more interesting. The chemical composition of shells does not vary as much as that of rocks. Their major component is calcium carbonate, and the results are relatively easy to predict, even though other substances like conchiolon, phosphates, silicates and pigments may be present. The external conchiolon layer should be removed during cleaning. Shells with smooth surfaces are not suitable for this work, but *Murex* and oyster types are excellent (3). The diversity of effects with rocks is larger, as the choice in chemical composition is wider. The main host rocks are given in Box 4 and the important features of each group are described in the next paragraphs.

**Granite, feldspathic and quartz rocks:** “acid rocks”, their largest component being silica,  $\text{SiO}_2$ . They appear to stimulate the crystallization of ionic salts, as their chemical composition is similar. They do not react with the solutions, and although they are very hard, dense and non-porous, crystals will attach strongly to the surface. For these reasons they are ideal for estimating the concentration of the solution after crystallization (3).

## Box 4

### HOST ROCKS

#### Magmatic or igneous rocks

Intrusive	Extrusive
<b>granite, feldspathic and quartz rocks</b> (silica-rich rocks)	<i>basalt, pumice, pyroclastic rocks, effusive rocks, volcanic tuffs</i>

#### Sedimentary rocks

**sandstone, arenites, conglomerates, limestone**

#### Metamorphic rocks

**quartzite (formed from arenites) and marble (calcite)**

#### Artificial Rocks

**concrete, cement**

*(Note: best samples in bold).*



*Basalt*, if very fine grained, hard and dense, is risky to use. Crystals formed on its surface fall apart easily. The major components are pyroxene (iron, magnesium and calcium silicates), olivine (iron and magnesium silicates), plagioclase (feldspar) and other minerals such as magnetite and biotite. Dangerous reactions for these rocks. are described in the next section.

*Pumice* is too light, floating at the liquid surface, and therefore not suitable for crystal growth.

**Pyroclastic rocks, effusive rocks and volcanic tuffs:** very good, except for calculations of the final concentrations of solutions, as they are generally very porous, retaining the solvent (mass balances are described in reference 3). Dark samples enhance transparent crystals.

**Sedimentary rocks, like sandstone, arenites, conglomerates and limestone,** react like shells. The products of decomposition for limestone will be formed in large amounts, which will then be difficult to wash off the crystals. The results might not be perfect (see section 2.3).

**Metamorphic rocks** are divided into two groups: **marble** behaves like sedimentary rocks, and **quartzite** like quartz rocks (see section 2.3).

**Artificial rocks:** concrete and cement are reactive in acidic solutions, with the same effects as sedimentary rocks (see section 2.3).

### 2.3 The main reactions (or interactions) of host surfaces with solutes.

These reactions are mainly of two types: oxidation-reduction and acid-base reactions. Habit modifiers are also included in this section.

### **2.3.1 Oxidation-reduction reactions:**

Metallic crystals mentioned in Boxes 2 and 3 are formed on metal wires by redox reactions. These popular “metal trees” are well-known examples producing Cu, Pb, Ag and Sn crystals, but are not included in the video.

**Copper crystals:** solutions of copper sulfate/Zn(s)

$$E^{\circ} (\text{Cu}^{2+}/\text{Cu}) = 0.34 \text{ V and } E^{\circ} (\text{Zn}^{2+}/\text{Zn}) = -0.76 \text{ V}$$

**Lead crystals:** solutions of lead nitrate/Zn(s)

$$E^{\circ} (\text{Pb}^{2+}/\text{Pb}) = -0.126 \text{ V and } E^{\circ} (\text{Zn}^{2+}/\text{Zn}) = -0.76 \text{ V}$$

**Silver crystals:** solutions of silver nitrate/ Cu(s), copper coins or Hg(l).

$$E^{\circ} (\text{Ag}^{+}/\text{Ag}) = 0.90 \text{ V and } E^{\circ} (\text{Cu}^{2+}/\text{Cu}) = 0.34 \text{ V}$$

$$E^{\circ} (\text{Ag}^{+}/\text{Ag}) = 0.90 \text{ V and } E^{\circ} (\text{Hg}^{2+}/\text{Hg}) = 0.854 \text{ V}$$

All these reactions are enhanced if shown with amplified under a microscope. They are good demonstrations for oxidation-reduction reactions, Latimer diagrams and the reduction potentials of the elements (3).

### Other redox reactions:

Chemicals that can act as oxidants for iron ( $\text{NaNO}_3$ ,  $\text{NaBrO}_3$ ,  $\text{NaClO}_3$ ) hosted on volcanic rocks **undergo dangerous side redox reactions** with liberation of bromine, chlorine, etc. (2). These and other combinations of this type are, therefore, not performed, even though the crystals may be interesting to study (10).

Redox reactions can occur when schönites of cobalt and copper are mixed together (2), but apparently they depend not on the host but on the solutes themselves.

### 2.3.2 Acid-Base reactions:

Calcium carbonate from shells, limestone, marble and sedimentary rocks will react strongly with acid solutions: metal salts acting as Brönsted acids in water will produce strong bubbling of carbon dioxide (reaction 1):

#### Reaction 1

$\text{M}=\text{Cu, Cr, Al, Fe, Zn, Ni, Co}$



These reactions will compete with crystal growth, partially decomposing the solute. In some cases, concentrations should be higher to compensate for this effect. A few examples can be observed on the video: chromium alum will show octahedral crystals, standing proudly over a greenish microcrystalline substance covering the shell (Table 1, 14); hydrated copper sulfate will form, as a side product, a layer of small brilliant bluish-green needles (Table 1, 7). For ammonium copper



double sulfate (copper schönite) (Figures 19 and 20), the side reactions are even stronger: for lower concentrations of the double salt, this decomposition will completely suppress growth, but the results will be rewarding, with the surface looking like fluffy green velvet! Very often these effects will make the sample even more attractive.



Figure 19. Copper schönite  $(\text{NH}_4)_2\text{Cu}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  (2) without decomposition on quartz.

For very delicate shells or corals, neutral or basic solutions are required, as acid solutions might completely destroy them. Let us take the example of double sulfate schönites  $\text{M}_2^{\text{I}}\text{M}^{\text{II}}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ : magnesium ( $\text{M}^{\text{II}}$ ) (Table 1, 18) should replace zinc(2) for transparent compounds, and potassium ( $\text{M}^{\text{I}}$ ) will be better than ammonium; potassium sodium tartrate (Table 1, 11, Rochelle salt; Figure 3) like any other basic solutions will preserve these shells.

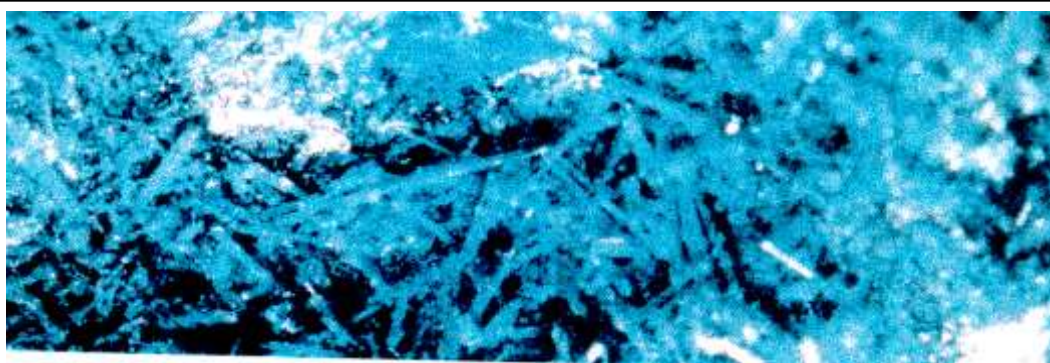


Figure 20. Copper schönite  $(\text{NH}_4)_2\text{Cu}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  (2) with decomposition. A piece of concrete was used as host rock. The sample looks like velvet covered with small crystals of unknown composition (seen in detail below).

### **2.3.3 Habit modifiers**

Some rocks can also act as strong habit modifiers (see section 2.4). Ammonium and potassium dihydrogen phosphate (KDP) will grow as whiskers on volcanic rocks (Table 1, 5); crystal growth will be stimulated by granite for the same solutes (Table 1, 6), and all the other rocks will fall between these two extremes, for the two compounds. Occasionally, inclusions of iron compounds at the surface of quartz rocks, can also induce a moderate change in habit for these compounds (the crystals will become needle-like and thinner).



Rocks will also modify crystals of Rochelle salt: this compound changes its habit if certain modifiers (e.g.  $\text{Cu}^{2+}$  ions) are added to the solution (10), developing into long columns (Figure 21). This effect can be seen to some extent on samples grown in the presence of volcanic rocks (sample 12, Table 1). However, marble or other rocks containing calcium carbonate apparently cancel this effect and the crystals in these conditions become more tabular. A slight increase in the pH of the solution might be the cause of this modification.



Figure 21. Potassium sodium tartrate  $\text{KNa}(\text{C}_4\text{H}_4\text{O}_6) \cdot 4\text{H}_2\text{O}$ , Rochelle's salt, with copper sulfate adsorbed on the surface. This sample shows habit modifications described in the literature (10). These crystals are called “aqua marina”, for their faint blue color (2).

**Some reactions are very important for cleaning the samples:** commercial sodium hypochlorite solutions are best for removing dirt and organic residues from rocks and shells, when a steel brush is not enough to solve the problem. Bleaching solutions containing hydrogen peroxide are not efficient and they are acidic. Acid solutions should be avoided, as they will react strongly



with some of the materials: marble, for instance, will be attacked by hydrochloric acid, and its surface will become too smooth to be suitable for crystal growth.

## **2.4 The Technique**

The methods of crystallization can be classified as shown in Box 5. Highly specialized ones were omitted (1, 8, 9, 20) and only those relevant to high schools and suitable for demonstrations in any classroom or ordinary laboratory are indicated. This classification is much simpler than, but compatible with, the more detailed version given by Hulliger (1). Comments on this subject and a few examples are included in a separate publication (3, 6d, 21).

The general preparation is given in Box 6. The solubility of most of these substances increases with temperature. Evaporation of the solvent is therefore not crucial, and slow cooling will be enough to produce the desired results (Box 5, method **b2**). If this fails, controlled evaporation should be tried (method **b1**), making some holes in the plastic foil covering the beakers. According to the classification in Box 5, this will be a mixed process, c. The evaporation method alone is not the best in these cases, as it leads to the formation of too many small crystals at the liquid surface. These will fall down over the rock, damaging the single crystals being formed below. Moreover, if too much solvent is evaporated, the rock may no longer be covered by the solution and these small crystals at the liquid surface will deposit on its surface, forming unsightly stratified layers.

Compared with the “thread method” for crystal growing described by Holden (10), the “on the rocks” method is much easier and more straightforward, because it is not necessary to provide seeds prepared in a separate experiment. The crystals produced are frequently much more transparent and perfect than those obtained with the thread method, even if the chances of contamination with impurities are higher. The heavy and steady rock is likely to avoid vibrations, which are connected

with the appearance of some turbidity. Another advantage of our method is the more regular form of the crystals obtained, as the growth is not hindered as much as it is under other conditions. Alum frequently shows a tabular habit when grown in the bottom of a beaker by evaporation; in our case it will form a perfect polyhedron (an octahedron truncated by a cube), as it can grow in almost all directions. The same is true for chromium alum and some other compounds.

### **Some practical advice:**

**Glass should always be used** to prepare the saturated solutions. Never use metallic containers, as they might induce strong redox reactions with the solutions! Certain compounds, like potassium ferricyanide, potassium dihydrogen phosphate, KDP, and the corresponding ammonium salt are so fast growing and hard that they might break the glass. For crystal growth only, heat-resistant plastic beakers are an attractive alternative in these cases, even though more expensive (not all types of plastic are suitable for crystal growing; test first with a small amount of solution).

Keep the crystals growing in a quiet place, where the ambient temperature is constant and there are no vibrations. Don't disturb them, and use a pencil flashlight for observation, if required.

### **What can go wrong with your experiments?**

If you have a limited time to get your final results, especially during summertime, cooling in a refrigerator may be required: the use of a water bath, to provide thermal inertia and avoid sudden changes in temperature, will be very important to control the cooling rate. Be sure you don't freeze the solution, otherwise you will not get any results.

## **Box 5**

### **CRYSTALLIZATION METHODS**

#### **PHYSICAL METHODS**

- a. Without solvent - change of state:
  - a1. Melting/solidification
  - a2. Sublimation/condensation from gaseous phase
- b. With solvent(s):
  - b1. Solvent evaporation
  - b2. Dependence of solubility on temperature
  - b3. Addition of a precipitating agent
  - b4. Freezing of the solvent
- c. Mixed processes

#### **CHEMICAL METHODS**

- d. Oxidation-reduction reactions
- e. Double ionic exchange or metathesis reactions
- f. Addition reactions (formation of double salts, alums and schönites)
- g. Others



If you can't get crystals, or if they are too many and too small, you should correct the initial concentration of the solution and adjust it to the place where you are growing your crystals. In the first case, you should evaporate more gently, to increase the concentration, or dissolve more substance. In the case of many small crystals your concentration is too high: go back to the beginning and add more solvent to decrease the degree of supersaturation.

If your solute is difficult to dissolve, grind it before you start the preparation of the solution. If it is impure, prior filtration will be required.

Slightly hygroscopic or deliquescent crystals should not be prepared on porous rocks that might retain small amounts of solvent. Those compounds showing strong hygroscopicity are not suitable for crystal growth, as they cannot be preserved.

If the rocks are too big, use a hammer to break them, but don't forget to wear your safety goggles and cover the rock with a cloth to avoid problems caused by flying fragments. **Do not forget the safety rules: always wear a white coat, goggles, and gloves when working with toxic substances. Use a powder mask if you need to grind the solutes. Do the preparations under a hood or in a well-ventilated room. Avoid breathing the vapors. If you are working with a class in the laboratory, making several preparations simultaneously, limit those with toxic substances to one or two.**

The most important data to record during an experiment are indicated in Table 2, enabling calculation of the final concentration of the solution and representation of the initial and final conditions in a solubility diagram. Equilibrium solubility data can be taken from references 22-24. Some of the results obtained are discussed in the literature (3).

## **Box 6**

### **How to prepare the crystals (“synthetic minerals-” or “jewels”)**

Make a saturated solution in a beaker at about 60-70 °C (water-soluble compounds).

Put the rock fragment or shell carefully inside the beaker, immersing it completely.

Cover the beaker with plastic foil to reduce evaporation. Let it cool undisturbed as slowly as possible (inside a warm water bath).

After 1-2 days, make some holes in the foil to allow evaporation of the solvent.

Wait for a few days.

Remove the rock with the crystals (any rock or shell for copper sulfate).

Recover the solution, by filtration, for other preparations.

Wash the crystals gently in very cold water and afterwards in ethanol.

Dry the crystals in cold air and protect them with a layer of nail polish.

Keep them inside a transparent plastic or glass box sealed with silicone wax. Protect from light, dust, heat and humidity.

**Table 2 - Data during the experiment**

Start Date:	End Date :
Mass of beaker (empty)/g	Total mass (beaker, solution, rock and crystals)
Mass of solute 1/g-	Mass of water lost by evaporation
Mass of solute 2/g-	Mass of crystals grown out of the host rock/g
	Mass of crystals on host rock/g
Mass of host rock or shell/g	Total mass of crystals/g
	Final concentration of solution
Classification of rock or shell	Period of crystal growth
Volume of water /ml	Was there any reaction with the host rock or shell?
	Why?
Volume of solution/ml	Dimensions of crystals, maximum
	minimum
Total mass/g (beaker + solution + host rock or shell)	Crystal system
Temperature for dissolution/ ° C)	Final solution temperature/ ° C (interruption of crystal growth)
Room temperature / ° C	Room temperature /°C
pH	pH



Make a plot of the solubility as a function of temperature for the compound you are studying (see the data in references 22-24). Represent the initial and final points of your preparation. Make some comments on your final values for the composition of the solution after crystal growth.

Your crystals look like minerals and gems. Suggest similarities, and indicate how you can distinguish them from the false ones.

#### **2.4.1-The Mechanism of Crystal Growth**

The chances of stimulating crystal growth are higher with this method than with conventional methods. This is in agreement with reports in the literature on crystallization of proteins using **heterogeneous nucleation** by minerals (25, 26): the degree of supersaturation required for growing protein crystals by epitaxy on minerals is lower than by conventional methods. (The degree of supersaturation, GS, is defined as  $GS=(S'-S)/S$ , where  $S'$  is the total concentration of the solute and  $S$  is the concentration of a saturated solution in equilibrium at each temperature (9, 20)). The same authors also observed unusual habits and even changes in the crystallographic system for some of the samples produced. Most of our host rocks and shells are composed of several or even many minerals, but the results for each class of minerals have not been systematically quantified. However, although our observations are purely qualitative, the interpretation is pretty much the same and the main stages for crystal growth in our case are the following:

**1. Nucleation.** Homogeneous nucleation is described in references 1, 9. The rock will first accelerate nucleation, by providing a large quantity of particles in suspension; then many small ledges, pits and holes will be provided by its rough surface, acting as nests for these nuclei, stabilizing the microscopic aggregates and leading to the formation of the first unit cells.

**2. Growth.** The crystals will start to grow, repeating the characteristic pattern of the substance in an orderly way; strong interactions between the rock components and the solute in solution will very likely increase the rate of accretion.

**3. Termination of growth.** Equilibrium is reached and growth stops, unless the conditions are modified.

The detailed mechanisms of nucleation and crystal growth are certainly complex and require thorough and systematic investigation, part of which we are undertaking. It seems that crystal growth (after nucleation) of inorganic substances is much more enhanced than organic substances when using rocks or shells. On the other hand, potassium sodium tartrate (Fig. 3), for example, appears to grow at the same rate with or without the rock, this being in agreement with the organic character of the tartrate anion. For this compound, however, a stimulation of growth was also clearly observed in the presence of oyster shells (Table 1, 11 and in many other preparations). It is therefore premature to make general conclusions on this subject by simply dividing the substances into organic or inorganic. Each system formed of solute/host/solvent is a very complex one and should be studied systematically to draw conclusions on which mechanisms of crystal growth are favored. This is, however, difficult, since each particular rock is different and different results may be obtained even when all other conditions are kept exactly the same.

### 3. The Concepts

Many basic concepts can be introduced to students using this methodology, as listed in Box 7, during and after the preparations. Our point of view is always that of a chemist, trying hard to attract young students, especially those who have inadequate preparation in chemistry. We found, indeed, that our beautiful crystals were powerful allies for that purpose.

Some examples of this methodology have been published or submitted (5-7) including applications to aluminum and copper chemistry, with the synthesis of several compounds,

### **Box7**

#### **LIST OF CONCEPTS**

**Physics, Chemistry, Material Sciences, Mineralogy and Geology**

**Introduction to Solid State Chemistry**

- Reactions in aqueous solution: acid-base, redox, precipitation, and complexation**
- Crystallization techniques, filtration, and other related techniques**
- Solubility diagrams and temperature dependence, chemical equilibrium for precipitation reactions, calculation of concentrations in solution, mass balances**
- UV-VIS absorption spectra, Beer's law**
- Intermolecular forces and solubility**
- Characterization of solids, lattice enthalpies. Solid solutions**
- Anhydrous and hydrated salts. Hygroscopicity, efflorescent and deliquescent substances**
- Enthalpies of formation, dissolution, thermodynamics of dehydration processes**
- Crystal systems, crystal habit, habit modifiers**
- Chemistry of the elements**
- Synthesis and characterization of compounds**
- Determination of melting points for pure compounds**

analytical characterization, preparation of crystals, and parallel experiments on the chemistry of these elements and their compounds. We kept the technique very simple to be able to attract a wider audience, especially high schools with scarce resources for conducting experiments. Finally, we



believe that linking the study of chemistry with other fields of science will give the students a better preparation for solving future professional problems. To our knowledge, no other authors have reported this method in the field of education in chemistry and related fields, except for the references on protein research and a few others mentioned in the next section. Heterogeneous nucleation does, however, seem quite promising for preparation of crystals for X-ray structural analysis, and its mechanism deserves further and systematic research to be fully understood (27). We are also trying to develop some scientific research projects to solve specific problems arising from the application of this methodology, in partnership with high school teachers at their schools.

#### **4. Concluding remarks**

We started this work in December 1993, during a General Chemistry lecture for first year students in IST, trying to arouse their interest in the classification of solid structures: a piece of a quartz rock was immersed in a supersaturated solution of hydrated copper sulfate, to induce faster crystallization. Carried away by the wonderful results obtained, we made a rapid literature search just looking for large crystals in the Merck Index (13), and using our common knowledge of well-known compounds for crystal growth, like the alum family. Just one year later, after publishing the first paper (6), we found that, except for the rock in our hands, we were following the steps given by Holden and Morrison in "Crystals and Crystal Growing" (10), sharing the same enthusiasm, and in some cases rediscovering almost identical recipes. At that time we were unaware of their work and as we were on our own, neither copying nor following anyone, we found the schönites family and other compounds, and it was amazing later on to read the first quotation in this book:

*"I would remind you then that when we want to find out anything that we do not know, there are two ways of proceeding. We may either ask somebody else who does know, or read what the most learned men have written about it, which is a very good plan if anybody happens to be able to answer our question; or else we may adopt the other plan, and by arranging an experiment, try for ourselves."*

*C. V. Boys, Soap Bubbles and the Forces Which Mould Them (a Science Study Series book).*

Since then, crystallization "on the rocks" has been included in many chemistry experiments at high school and on basic university courses in General Chemistry. We adapted some of Holden's recipes to our method and started small research projects. The results have been encouraging, attracting students' interest beyond all expectations, and very soon we were surrounded by students and teachers of chemistry, physics, mineralogy and geology at both high schools and universities, and also by an increasing number of mineral and shell collectors and artists.

We cannot identify those who tried this method first, but Mother Nature has been doing it all the time and the natural gems and minerals surrounding us are the beautiful products of her continuous work. The ability to simulate the genesis of minerals in a very simple way, and the arresting beauty of the large single crystals produced, are probably the major attractions of this method. Perhaps this is the reason why it can bring together children and adults, students, teachers, and people with many different kinds of interests. Many have taken advantage from this. Rough imitations of minerals, prepared in this way, are sold here and there all over the world. Volcanic rocks with deep blue crystals of hydrated copper sulfate can be easily recognized in mineral collections, and are sold to tourists in the beautiful city of Taormina, Sicily, as natural minerals from Mount Etna's eruptions. Potassium alum doped with a yellow dye, in its most common habit (octahedral truncated by cubic faces) is also sold as natural sulfur, probably exploiting its similarity



with the prismatic orthorhombic crystals of that element (Figures 22). Small crystals of potassium dichromate on dark volcanic rocks are another example from these collections (Figure 23). Many other imitations by this and other methods are cited in the literature (28).



Figure 22. Potassium alum doped with tartrazine is sold as native sulfur in mineral collections from Etna (2).

Recently a few crystal-growing sets have been sold as children's toys, with for safety reasons a very limited range of chemicals, and much larger units for education in chemistry in high schools and universities have been designed by our research group.

There are bound to be those who will become addicted to this methodology, as a hobby, or even a business. They will have a lot of fun searching for different rocks and shells, trying to find compounds of different colors and crystallization systems that will grow single crystals easily. The range of compounds one might combine, plus all the different types of rocks and shells, gives an enormous number of possible combinations, increased by the reactions



between the solutions and their hosts. Bearing in mind that rock collections are currently in fashion, "On the Rocks" crystallization is likely to become widespread, perhaps equaling in popularity Holden and Morrison's classical technique (10), being even easier to perform and with greater variety. Some people will do these experiments at home, where accidents are liable to happen if **safety rules are not strictly followed**. Toy distributors are very careful in the choice of products they include in their sets, but however many safety rules they include, they will never overcome people's curiosity or prevent them from trying other products, just to see if they work! So the best way to avoid poisoning by chemicals or other problems caused by long exposures, such as dermatitis or allergies, is to get full and accurate information. The right place for this is at school. This was our idea when we started our teaching program for high school teachers and writing these notes. Be careful when starting this as a hobby, dear reader, have a lot of fun and don't get a "hangover"!



Figure 23. Other compounds sold as minerals from volcanic eruptions (2).

**Suggestions for some “aperitifs” and a toast to Louis Pasteur!**



**Follow the instructions given in Box 6**

**1 – 50-56 g of pentahydrated copper sulfate dissolved in 100 ml of water (any host). Wait for one week.**

**2 – 37-45 g of potassium dihydrogen phosphate or ammonium dihydrogen phosphate dissolved in 100 ml of water (granite). Wait for one week.**

**3 – 26-30 g of hydrated potassium alum dissolved in 100 ml of water (any rock or shell). Wait for one week.**

**4 - 50 g of potassium ferricyanide dissolved in 100 ml of water (any rock or shell). Wait for one week.**

**5 – 130-139 g of Rochelle’s salt dissolved in 100 ml of water (any rock or shell; oysters!).**

**Wait for two or three weeks. Seed the solution with a few small crystals if crystallization has not begun within three weeks.**



**Cheers!!!!**



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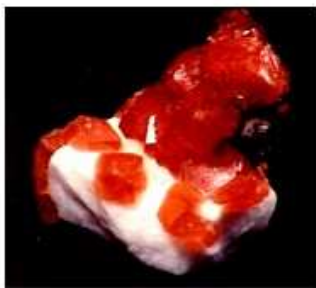
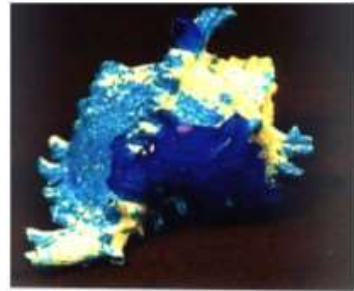


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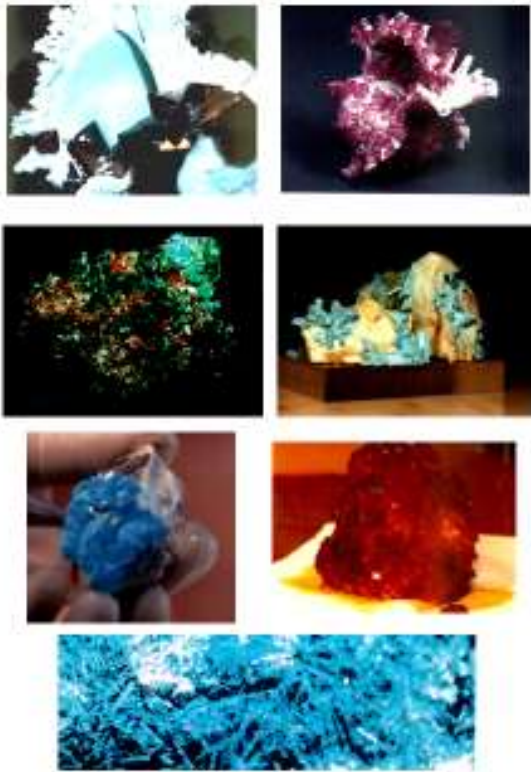
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**“On The Rocks...”  
Crystallization on  
Rough Surfaces**

by Clementina Teixeira



**“On The Rocks...” Crystallization on  
Rough Surfaces  
Clementina Teixeira  
Video n.º2**

Universidade Técnica de Lisboa  
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Centro de Química Estrutural  
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