

[o.m13.p11] On the rocks... crystallization on rough surfaces. new applications. C. Teixeira, *Centro de Química Estrutural, Instituto Superior Técnico, Av. Rovisco Pais 1049-001 Lisboa, Portugal. Email: pcclementina@alfa.ist.utl.pt*

Keywords: giant crystals, crystal growth on rocks, inorganic group chemistry.

In most part of textbooks for general education in Chemistry, the subject of crystallization and crystal growth is very often neglected¹. However, these can be very effective techniques for catching the young student's interest in many fields of Science and even in Arts. In this presentation, crystal growth stimulated by rocks, shells and other rough surfaces is combined with aluminum chemistry: first, potassium alum is synthesized from aluminum foil as recommended in the literature². Large single crystals of the pure compound are afterwards produced, allowing it to crystallize on rough surfaces of chosen composition. Different combinations with other alums (chromium alum, Figure below, ferric alum, etc.) dyes and metal salts are also prepared, including solid solutions and heterogeneous mixtures. Solubility diagrams are used for mass balances during these preparations, enabling the calculation of the concentration of the final solutions. Other experimental methods are used to confirm the results. A list of tests for aluminum chemistry is also included as an extension for group chemistry studies.



Figure: Chromium alum crystals drawn by Cristina Fontoura Carvalhão. Crystals were grown on arenite by the "On the Rocks" method.

Acknowledgements: Ministério da Ciência e Tecnologia, Program Ciência Viva. Ministério da Educação, PRODEP II FOCO.

[1] Teixeira, C. "Os Cristais no Ensino e Divulgação da Química.", Colóquio Ciências, Fundação Calouste Gulbenkian, (2000), 25: 18 - 36. References cited therein.

[2] Summerlin, L.R., Borgford, C.L. Ealy Jr., J.L. "Demonstrações de Química", Sociedade Portuguesa de Química, 2nd ed., (1996) vol.2 : 65-66.

[3] Teixeira, C. "Rochas Ornamentais e Minerais Sintéticos", Brincando com a Cor, Química, Boletim da Sociedade Portuguesa de Química, (1999), 74: 55-58. References cited therein.

[o.m13.p12] Investigating the Configuration at Vanadium in Highly Active Catalysts for Stereoselective Oxidations. I. Svoboda¹, M. Greb², J. Hartung², P. Schmidt², H. Fuess¹ ¹Fachbereich Material-wissenschaft, Fachgebiet Strukturforchung der TU Darmstadt, Petersenstraße 23, D-64287 Darmstadt, Germany. ²Institut für Organische Chemie der Universität Würzburg, Am Hubland, D-97074 Würzburg, Germany.

Keywords: vanadium, chirality, catalyst.

Vanadium(V) complexes are important catalysts in stereoselective syntheses. Our own interest in this field of research is related to the formation of chiral cyclic ethers from alkenols by catalytic transformations. Recently, we have discovered that the reagent combination of a tridentate imine ligand, an alkyl hydroperoxide and VO(acac)₂ leads to a highly efficient catalyst for the synthesis of 2,5-*cis*-, 2,4-*trans*- and 2,3-*trans*-disubstituted tetrahydrofurans from the corresponding alkenols.^[1] Particular noteworthy is the fact, that diastereoselective but not enantioselective transformations were observed, although enantio-merically pure auxiliaries were applied in catalysis.

In order to rationalize the significance of vanadium(V) complexes in the synthesis of tetrahydrofurans and to discover the stereochemical "leak" in the tetrahydrofuran synthesis, formation of vanadium(V) imine complexes from the individual components was investigated by ⁵¹V-NMR spectroscopy. The results were correlated with independently prepared vanadium imine complexes which were studied by X-ray diffraction. Coordination compounds with optically active auxiliaries afforded stereochemically pure vanadium(V) complexes. Thus, depending on the nature of the organic ligand, either the Λ or the Δ -configuration at vanadium was observed. If the same complexes however were prepared under reaction conditions which correspond to the tetrahydrofuran synthesis mentioned above, two ⁵¹V-NMR resonances were recorded for each vanadium(V) imine and for each derived alkyl peroxy complex. Although the origin of this stereodesintegrity is not known at the moment, our data provide important information, for further characterization of catalytically active vanadium(V) imine complexes and their alkyl peroxy derivatives complexes in solution.

[1] Vanadium(V)-Catalyzed Stereoselective Synthesis of Functionalized Disubstituted Tetrahydrofurans. J. Hartung, P. Schmidt, *Synlett* **2000**, 361–364.

[2] S. Drees, H. Fuess, M. Greb, J. Hartung, P. Schmidt, I. Svoboda, *manuscript in preparation*.